

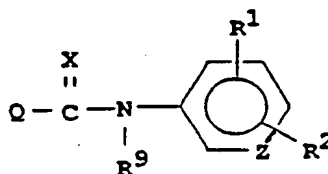


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| <p>(21) International Application Number: PCT/US91/09172</p> <p>(22) International Filing Date: 17 December 1991 (17.12.91)</p> <p>(30) Priority data: 638,720 8 January 1991 (08.01.91) US 657,702 19 February 1991 (19.02.91) US</p> <p>(60) Parent Applications or Grants (63) Related by Continuation US 638,720 (CIP) Filed on 8 January 1991 (08.01.91) US 657,702 (CIP) Filed on 19 February 1991 (19.02.91)</p> <p>(71) Applicant (for all designated States except US): E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).</p> | | <p>(72) Inventors; and (75) Inventors/Applicants (for US only) : HARRISON, Charles, Richard [US/US]; 137 Aspen Drive, Newark, DE 19702 (US). KRANIS, Kevin, Thomas [US/US]; 1413 North 12th Street, Reading, PA 19604 (US). STEVENSON, Thomas, Martin [US/US]; 103 Iroquois Court, Newark, DE 19702 (US).</p> <p>(74) Agents: COSTELLO, James, A. et al.; E.I. du Pont de Nemours and Company, Legal/Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).</p> <p>(81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), MC (European patent), NL (European patent), SE (European patent), US.</p> <p>Published Without international search report and to be republished upon receipt of that report.</p> |

(54) Title: ARTHROPODICIDAL CARBOXANILIDES

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(I)

(57) Abstract

Compounds of formula (I), wherein Q, X, R¹, R², R⁹ and Z are as defined in the text, including compositions containing said compounds and a method for using them to control arthropods.

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TITLE

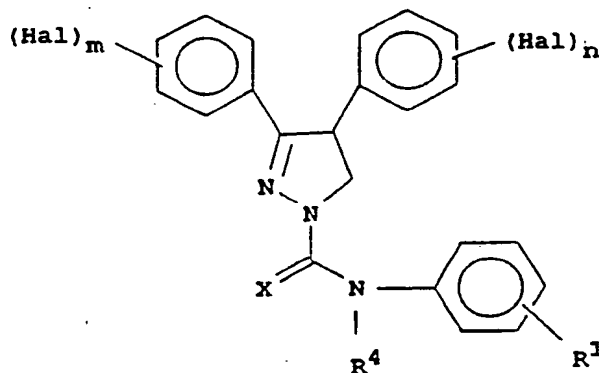
ARTHROPODICIDAL CARBOXANILIDES

BACKGROUND OF THE INVENTION5 Field of the Invention

This invention concerns arthropodically active carboxanilides, compositions containing them and a method for using them to control arthropods.

State of the Art

10 U.S. 4,070,365 discloses insecticidal pyrazolines of the formula:



15 wherein

R¹ is selected from the group halogen or optionally substituted alkyl; and

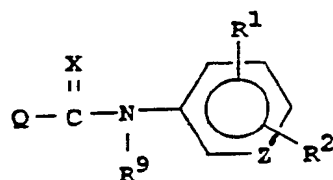
R⁴ is selected from the group H or alkyl.

WO 88/07994 discloses insecticidal indazole
20 derivatives. WO 90/07495 discloses insecticidal
semicarbazones. EP 3,913 discloses substituted
benzophenone hydrazones as insecticides.

SUMMARY OF THE INVENTION

25 The invention pertains to compounds of Formula I,
including all geometric and stereoisomers, agriculturally
suitable salts thereof, agricultural compositions

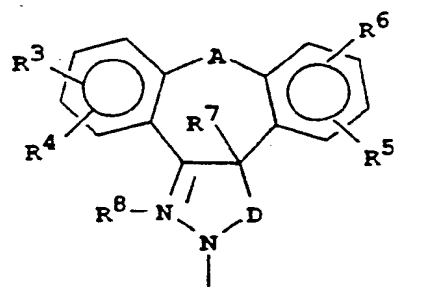
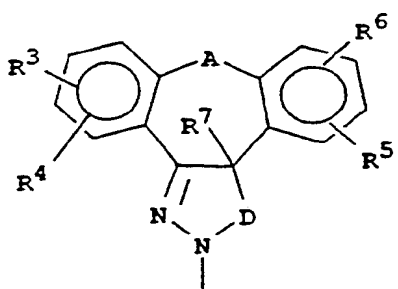
containing them and their use as arthropodicides in both agronomic and nonagronomic environments. The compounds are:



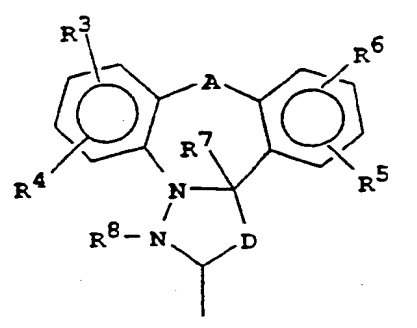
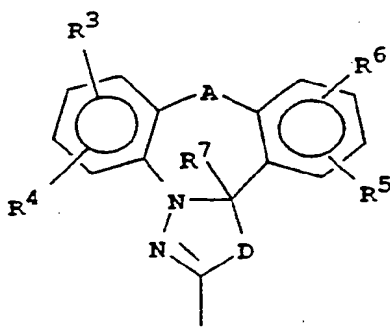
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wherein:

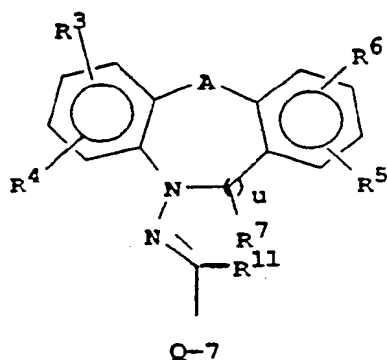
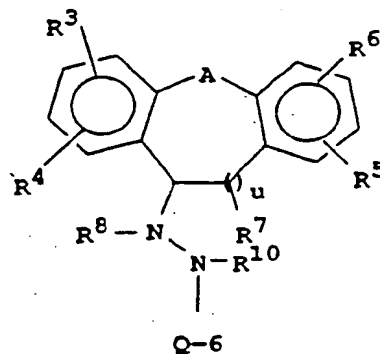
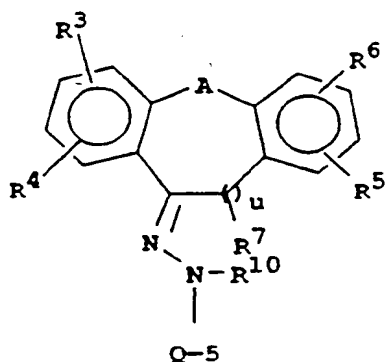
Q is selected from the group



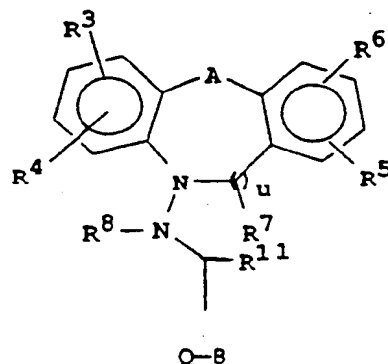
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3



and



- 5 A is selected from the group CH_2 , CH_2CH_2 , O, $\text{S}(\text{O})_p$, NR^{18} and $-\text{CH}_2(\text{G})-$; provided that i) when A is $-\text{CH}_2(\text{G})-$, either phenyl moiety can be attached to the CH_2 moiety and when A is CH_2 or $-\text{CH}_2(\text{G})-$, said CH_2 can be optionally substituted with 1 or
- 10 2 substituents independently selected from C_1 - C_2 alkyl, and ii) when u is 0, A is CH_2CH_2 or $-\text{CH}_2(\text{G})-$;
- D is selected from C_1 - C_2 alkylene optionally substituted with 1 to 2 substituents
- 15 independently selected from C_1 - C_2 alkyl;
- G is selected from the group O, $\text{S}(\text{O})_p$ and NR^{18} ;
- X is selected from the group O and S;
- Z is selected from the group N and CH;
- R^1 , R^2 , R^3 , R^4 , R^5 and R^6 are independently selected
- 20 from the group H, halogen, CN, SCN, NO_2 , R^{12} ,

OR¹², S(O)_qR¹², OSO₂R¹², C(O)R¹², CO₂R¹²,
C(O)N(R¹²)R¹³, SO₂N(R¹²)R¹³ and N(R¹²)R¹³; or
R¹ and R² or R³ and R⁴ or R⁵ and R⁶ when attached to
adjacent atoms can be taken together as OCH₂O,
5 OCF₂O, OCH₂CH₂O, OCH₂C(CH₃)₂O or OCF₂CF₂O to
form a cyclic bridge;

R⁷ is selected from the group H, CN, C₁-C₆ alkyl and
CO₂R¹²;

R⁸ is selected from the group H, C₁-C₆ alkyl, C₂-C₆
alkylcarbonyl, CO₂R¹² and C(O)N(R¹²)R¹³;

10 R⁹ and R¹⁰ are independently selected from the group
H, C₁-C₆ alkyl, C₂-C₆ alkoxyalkyl, CHO, C₂-C₆
alkylcarbonyl, C₂-C₆ alkoxycarbonyl,
C₂-C₆ haloalkylcarbonyl, C₁-C₆ haloalkylthio,
15 R¹⁴OC(O)N(R¹⁵)S-, R¹⁷(R¹⁶)NS- and benzyl
optionally substituted with W;

R¹¹ is selected from the group H, C₁-C₆ alkyl,
C₁-C₆ haloalkyl and phenyl optionally substituted
with W;

20 R¹² is selected from the group C₁-C₄ alkyl, C₁-C₄
haloalkyl, C₂-C₄ alkenyl, C₂-C₄ haloalkenyl, C₃-
C₄ alkynyl, C₃-C₄ haloalkynyl, C₂-C₆
alkoxyalkyl, C₂-C₆ alkylthioalkyl, C₂-C₆
cyanoalkyl, C₃-C₆ alkoxycarbonylalkyl, C₃-C₆
25 cycloalkyl, C₃-C₆ halocycloalkyl, C₄-C₇
alkylcycloalkyl, C₄-C₇ haloalkylcycloalkyl,
optionally substituted phenyl and optionally
substituted benzyl wherein the phenyl and benzyl
substituent(s) are 1 to 3 substituents
30 independently selected from W;

R¹³ is selected from the group H and C₁-C₄ alkyl;

R¹⁴ and R¹⁵ are independently selected from C₁-C₆
alkyl;

- R¹⁶ and R¹⁷ are independently selected from C₁-C₄ alkyl; or
R¹⁶ and R¹⁷ when attached to the same atom can be taken together as (CH₂)₅ or CH₂CH₂OCH₂CH₂;
5 R¹⁸ is selected from the group H, C₁-C₃ alkyl, CO₂R¹⁹ and SO₂R¹⁹;
R¹⁹ is selected from C₁-C₃ alkyl;
W is selected from the group halogen, CN, NO₂,
C₁-C₂ alkyl, C₁-C₂ haloalkoxy, C₁-C₂ alkoxy, C₁-
10 C₂ haloalkoxy, C₁-C₃ alkylthio, C₁-C₂ haloalkylthio, C₁-C₂ alkylsulfonyl and C₁-C₂ haloalkylsulfonyl;
p is 0, 1 or 2;
q is 0, 1 or 2; and
15 u is 0 or 1.

Preferred compounds A are those compounds of Formula I wherein:

- A is selected from the group S, CH₂CH₂ and
20 -CH₂(G)-;
D is C₁-C₂ alkylene;
R¹, R², R³, R⁴, R⁵ and R⁶ are independently selected from the group H, halogen, CN, R¹²,
S(O)_qR¹² and OSO₂R¹²;
25 R⁷ is CH₃;
R⁸ is H;
R⁹ and R¹⁰ are independently selected from the group H, C₁-C₂ alkyl, C₂-C₃ alkylcarbonyl and C₂-C₃ alkoxy carbonyl;
30 R¹¹ is selected from the group H and CH₃;
R¹² is selected from the group C₁-C₃ alkyl and C₁-C₃ haloalkyl;
R¹³ is C₁-C₂ alkyl;
R¹⁸ is H or CH₃;

p is 0; and
q is 0 or 2.

Preferred compounds B are those of Preferred A
wherein Q is Q-1.

5

Preferred compounds C are those of Preferred A
wherein Q is Q-2.

Preferred compounds D are those of Preferred A
10 wherein Q is Q-3.

Preferred compounds E are those of Preferred A
wherein Q is Q-4.

15 Preferred compounds F are those of Preferred A
wherein Q is Q-5.

Preferred compounds G are those of Preferred A
wherein Q is Q-6.

20

Preferred compounds H are those of Preferred A
wherein Q is Q-7.

Preferred compounds I are those of Preferred A
25 wherein Q is Q-8.

This invention also concerns an arthropodicidal
composition containing a compound of this invention with
a carrier therefor which is selected for the particular
30 arthropod to be controlled or contemplated end use. This
invention also concerns a method for controlling
arthropods by applying an effective amount of a compound
of the invention to them or to their environment.

In the above recitations, the term "alkyl", used either alone or in compound words such as "alkylthio" or haloalkyl", denotes straight chain or branched alkyl such as methyl, ethyl, n-propyl, isopropyl or the different
5 butyl, pentyl, hexyl isomers. Alkoxy denotes methoxy, ethoxy, n-propyloxy, isopropyloxy and the different butoxy, pentoxy or hexyloxy isomers. Alkenyl denotes straight chain or branched alkenes such as vinyl, 1-propenyl, 2-propenyl, 2-propenyl and the different
10 butenyl, pentenyl and hexenyl isomers. Alkynyl denotes straight chain or branched alkynes such as ethynyl, 1-propynyl, 3-propynyl and the different butynyl, pentynyl and hexynyl isomers. Alkylthio denotes methylthio, ethylthio and the different propylthio, butylthio,
15 pentylthio and hexylthio isomers. Alkylsulfinyl, alkylsulfonyl, alkylamino and the like are defined analogously to the above examples. Cycloalkyl denotes cyclopropyl, cyclobutyl, cyclopentyl and cyclohexyl.

The term "halogen", either alone or in compound
20 words such as "haloalkyl", denotes fluorine, chlorine, bromine or iodine. Further, when used in compound words such as "haloalkyl" said alkyl can be partially or fully substituted with halogen atoms, which can be the same or different. Examples of haloalkyl include $\text{CH}_2\text{CH}_2\text{F}$, CF_2CF_3
25 and $\text{CH}_2\text{CHFC1}$. The terms "halocycloalkyl" haloalkenyl" and "haloalkynyl" are defined analogously to the term "haloalkyl".

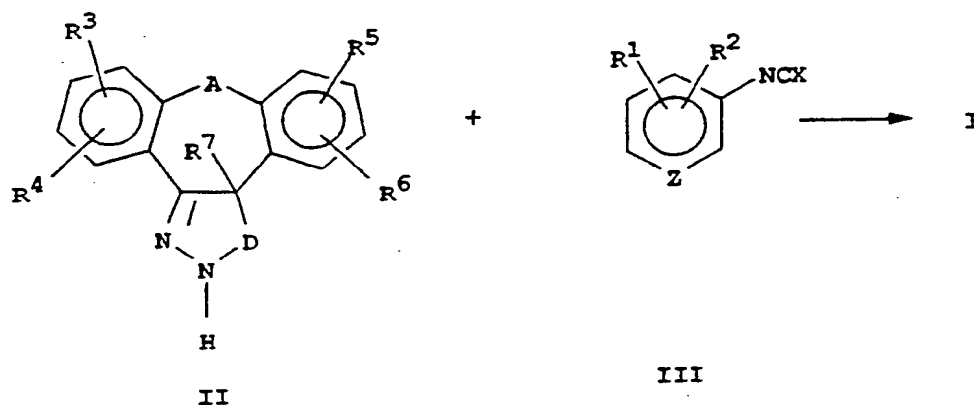
The total number of carbon atoms in a substituent group is indicated by the " $\text{C}_i\text{-C}_j$ " prefix where i and j
30 are numbers from 1 to 7. For example, $\text{C}_1\text{-C}_3$ alkyl-sulfonyl designates methylsulfonyl through propyl-sulfonyl; C_2 alkoxyalkoxy designates OCH_2OCH_3 ; C_4 alkoxy-alkoxy designates the various isomers of an alkoxy group substituted with a second alkoxy group containing a total

of 4 carbon atoms, examples including $\text{OCH}_2\text{OCH}_2\text{CH}_2\text{CH}_3$ and $\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_3$; C_2 cyanoalkyl designates CH_2CN and C_3 cyanoalkyl designates $\text{CH}_2\text{CH}_2\text{CN}$ and $\text{CH}(\text{CN})\text{CH}_3$; C_2 alkylcarbonyl designates $\text{C}(\text{O})\text{CH}_3$ and C_4 alkylcarbonyl includes $\text{C}(\text{O})\text{CH}_2\text{CH}_2\text{CH}_3$ and $\text{C}(\text{O})\text{CH}(\text{CH}_3)_2$; and as a final example, C_3 alkoxycarbonylalkyl designates $\text{CH}_2\text{CO}_2\text{CH}_3$ and C_4 alkoxycarbonylalkyl includes $\text{CH}_2\text{CH}_2\text{CO}_2\text{CH}_3$, $\text{CH}_2\text{CO}_2\text{CH}_2\text{CH}_3$, and $\text{CH}(\text{CH}_3)\text{CO}_2\text{CH}_3$.

DETAILS OF THE INVENTION

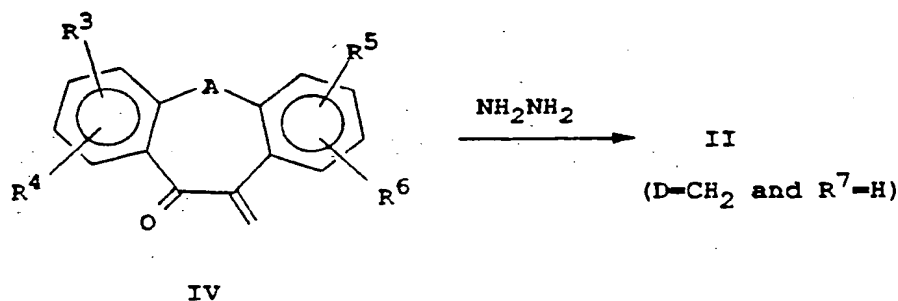
Compounds of Formula I, where Q is Q-1, can be prepared by the reaction of hydrazones of Formula II with aryl isocyanates of Formula III as shown in Scheme 1. Typical reactions involve the combination of equimolar amounts of II and III in conventional organic solvents including ether, tetrahydrofuran, methylene chloride, chloroform and benzene. The reaction can be run at temperatures ranging from about -20°C to 100°C with temperatures in the range of about -10°C to 30°C generally preferred.

SCHEME 1



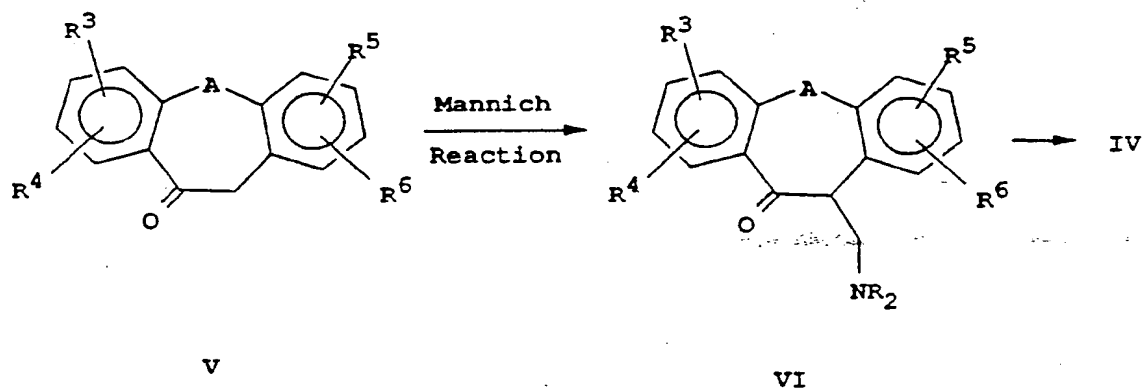
Substituted compounds of Formula II, where D is CH_2 and R^7 is H, can be prepared by the reaction of hydrazine

with an α,β -unsaturated ketone of Formula IV (or their precursors) by procedures well documented in the chemical literature (Scheme 2). For literature describing the synthesis of 3,4- and 3,5-disubstituted pyrazolines, which can be applied to the synthesis of compounds of Formula II, where R^7 is H, see U.S. Patents 3,991,073 and 4,070,365.

SCHEME 2

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Compounds of Formula IV can be prepared via a Mannich reaction followed by elimination of the dialkylamino group. Scheme 3 illustrates this transformation.

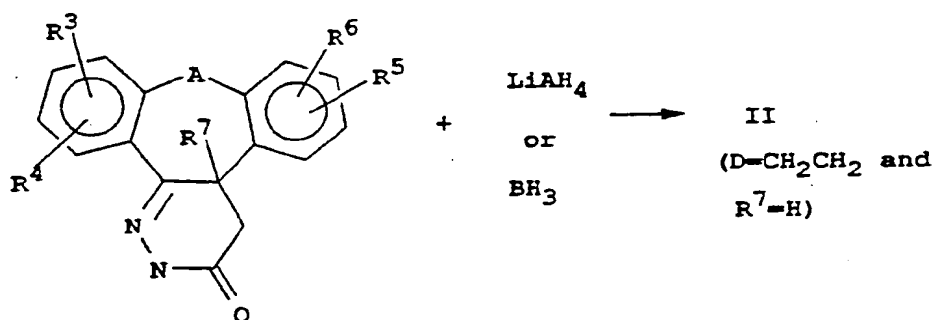
SCHEME 3

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The ketones of Formula V can be prepared by procedures taught in *Org. Prep. and Proc. Int.* **1984**, 16(5), 411-425, *J. Org. Chem.* **1978**, 43, 3698-3700 and references within.

- 5 Formula II compounds, where D is CH_2CH_2 , and R^7 is H, can be prepared by the reaction of Formula VII compounds with a reducing agent such as lithium aluminum hydride or diborane. The typical reaction involves the combination of an excess in molar amounts of the reducing agent (1.1 equivalents to 5.0 equivalents) with 1
10 equivalent of a Formula VII compound. Conventional aprotic organic solvents such as diethyl ether, tetrahydrofuran or 1,2-dimethoxy-ethane can be used. The reaction temperature can vary from 0°C to the reflux
15 temperature of the particular solvent being used and the reaction is usually complete in less than 24 hours. Scheme 4 illustrates this transformation.

SCHEME 4



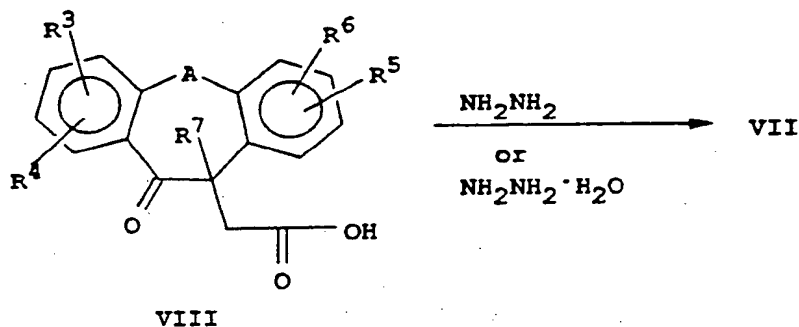
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VII

- Formula VII compounds can be prepared by the reaction of Formula VIII compounds with an excess (1.1 to 5.0 equivalents) of hydrazine or hydrazine monohydrate.
25 The reaction is conducted in an alcohol solvent such as methanol, ethanol, n-propanol, isopropanol, n-butanol and

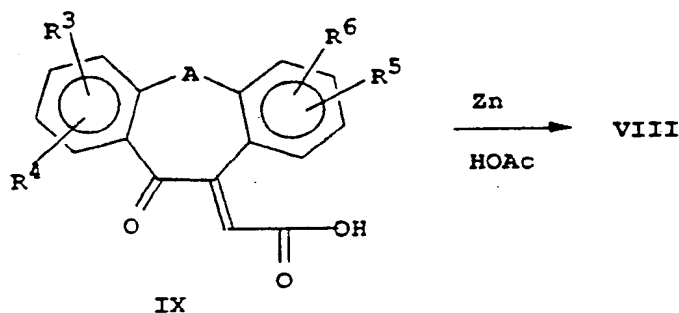
the like or acetic acid and the temperature is governed by the reflux temperature of the particular solvent. The reaction is generally complete in 24 hours. Scheme 5 illustrates this transformation.

5

SCHEME 5

Compounds of Formula VIII where R^7 is H can be prepared by the reduction of Formula IX compounds. This transformation can be effected by catalytic hydrogenation or more conveniently through the use of an excess (1.5 to 4.0 equivalents) of zinc in refluxing acetic acid as solvent. The reaction is usually complete in 24 hours. Scheme 6 illustrates this transformation.

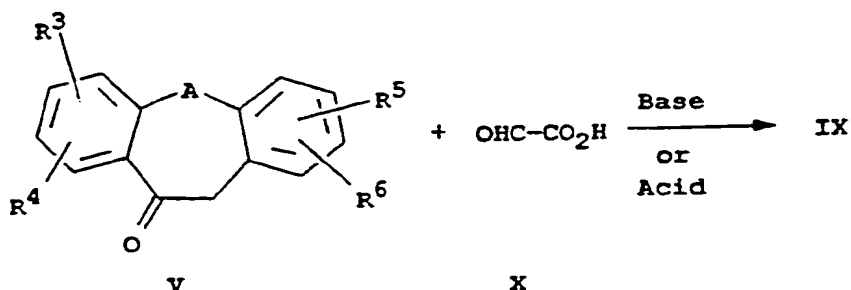
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SCHEME 6

Compounds of Formula IX can be prepared by the reaction of Formula V derivatives with Formula X

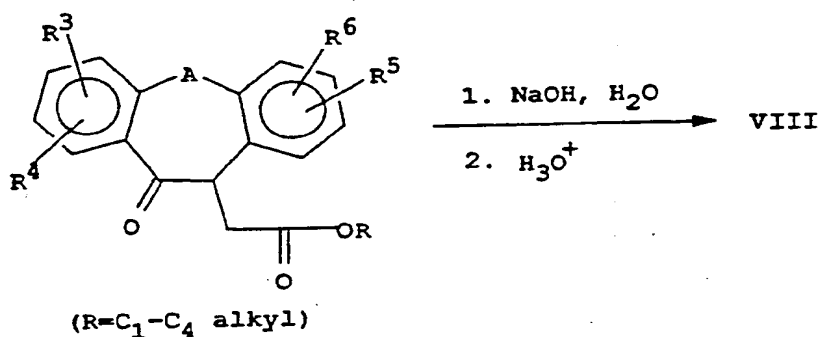
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compounds. This Aldol condensation reaction is illustrated in Scheme 7.

SCHEME 7

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Alternatively, compounds of Formula VIII can be prepared by the hydrolysis of Formula XI compounds by the transformation illustrated in Scheme 8.

SCHEME 8

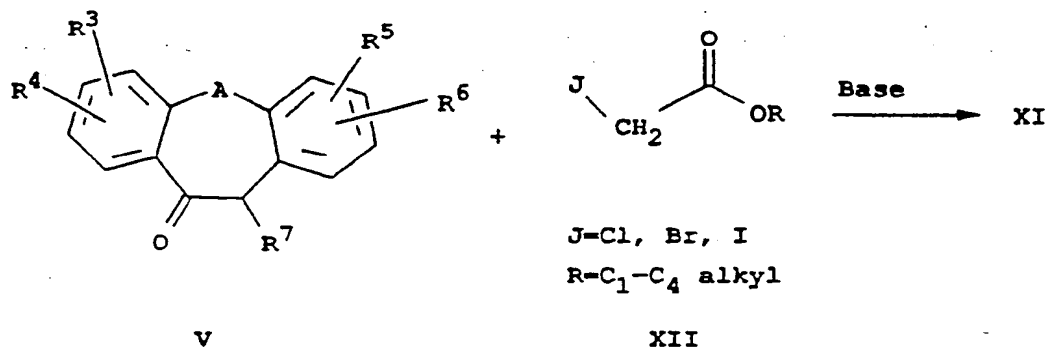
XI

Formula XI compounds can be prepared by the alkylation of Formula V derivatives with Formula XII compounds. The transformation can be accomplished by the reaction of equimolar amounts of Formula V and Formula XII compounds in the presence of a base such as an alkali metal, tertiary amine, metal hydride and the like in a conventional organic solvent such as ether,

15

20

tetrahydrofuran, 1,2-dimethoxyethane, dimethylformamide, dimethylsulfoxide, methanol, ethanol and propanol. The reaction is usually conducted at temperatures between 0°C and the reflux temperature of the solvent. The reaction is usually complete in 48 hours. Scheme 9 illustrates this transformation.

SCHEME 9

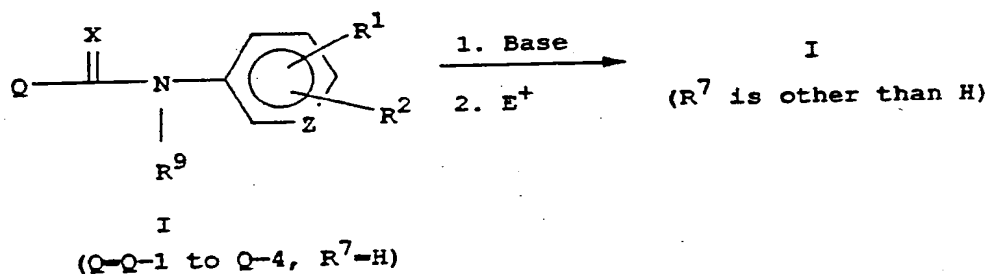
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Preparation of compounds of Formula I, where Q is Q-1 through Q-4 and R⁷ is other than H, can be achieved by metallation of the R⁷ position of Formula I (where R⁷ is H) followed by reaction with a suitable electrophile as depicted in Scheme 10. Metallation can be accomplished by deprotonation with a strong base, in a suitable solvent at temperatures ranging from -78°C to 100°C. Useful bases for this reaction include lithium dialkylamides such as lithium diisopropylamide and lithium tetramethylpiperidide, alkyl lithium reagents such as n-butyllithium and metal hydrides such as sodium hydride and potassium hydride. Deprotonation of compounds of Formula I, where R⁷ is H, may require two equivalents of base when R⁹ is H. The reaction can be conducted in conventional organic solvents and in certain instances a cosolvent is useful. Suitable solvents include diethylether, tetrahydrofuran, tetrahydropyran,

dimethylformamide, hexamethyl-phosphoramide, benzene, and the like. Suitable electrophilic reagents for the reaction with the metallated Formula I compounds include alkyl and substituted alkyl halides, alkyl
 5 chloroformates, acyl halides, isocyanates, dialkyl carbamoylhalides and related electrophiles which will be known to those skilled in the art. Scheme 10 illustrates this transformation.

SCHEME 10

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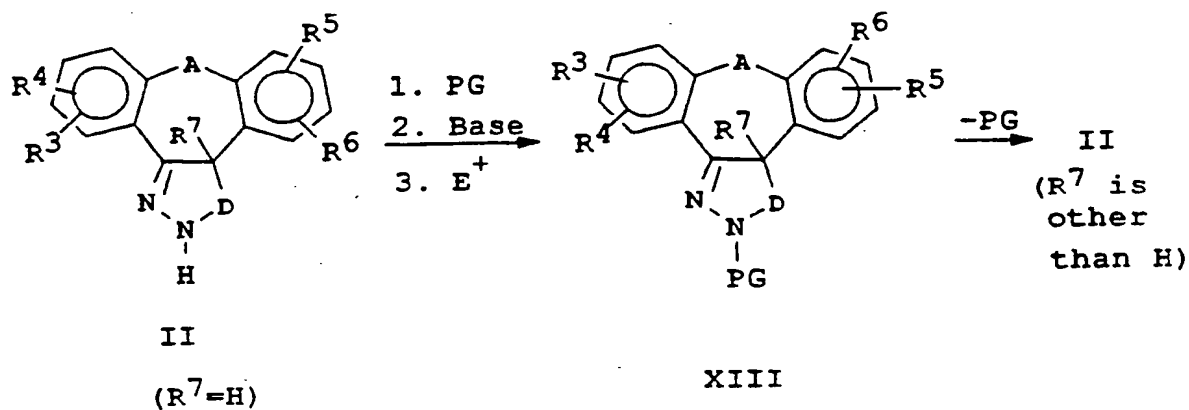


where E⁺ is an electrophile

An alternative procedure for introduction of the R⁷ substituent, which in certain instances may be preferred
 15 over that of Scheme 10 due to higher yields and/or greater ease of synthesis, proceeds via the intermediacy of Compound XIII, wherein the nitrogen has been derivatized with a suitable protecting group as shown in Scheme 11. Deprotonation with a strong base such as
 20 lithium diisopropylamide, typically in stoichiometric quantities, followed by reaction with any of the previously described electrophiles provides compounds of Formula XIII where R⁷ is other than H. Removal of the nitrogen protecting group provides the required Formula
 25 II intermediate. Nitrogen-protecting groups are well documented in the chemical literature, as are procedures for their preparation and cleavage. Examples include

acetyl, trifluoroacetyl, benzoyl, substituted benzoyl, alkoxy carbonyl, benzyl and substituted benzyl.

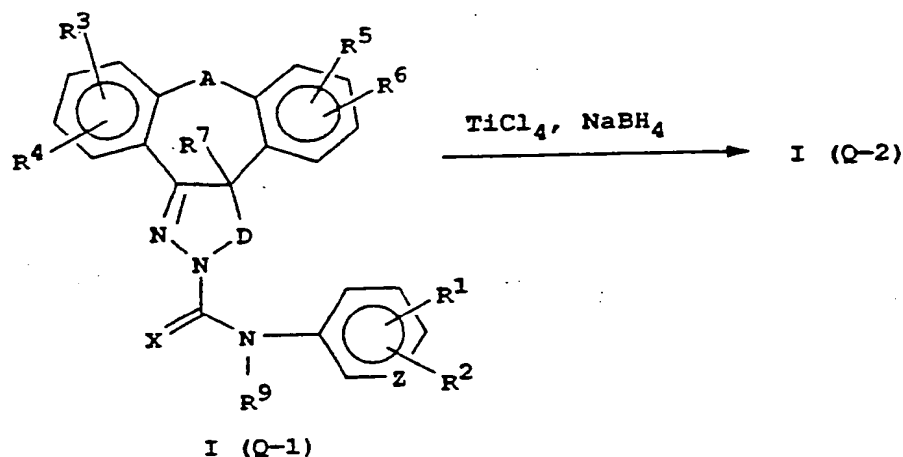
SCHEME 11



where E⁺ is an electrophile and PG is a suitable nitrogen-protecting group.

Compounds of Formula I (X=O) can be converted to compounds of Formula I (X=S) by means of thiating agents. Conversion of amides to thioamides is known in the art.

Compounds of Formula I (Q-2) can be prepared by a titanium tetrachloride/sodium borohydride reduction of Formula I (Q-1) compounds. Scheme 12 illustrates this transformation.

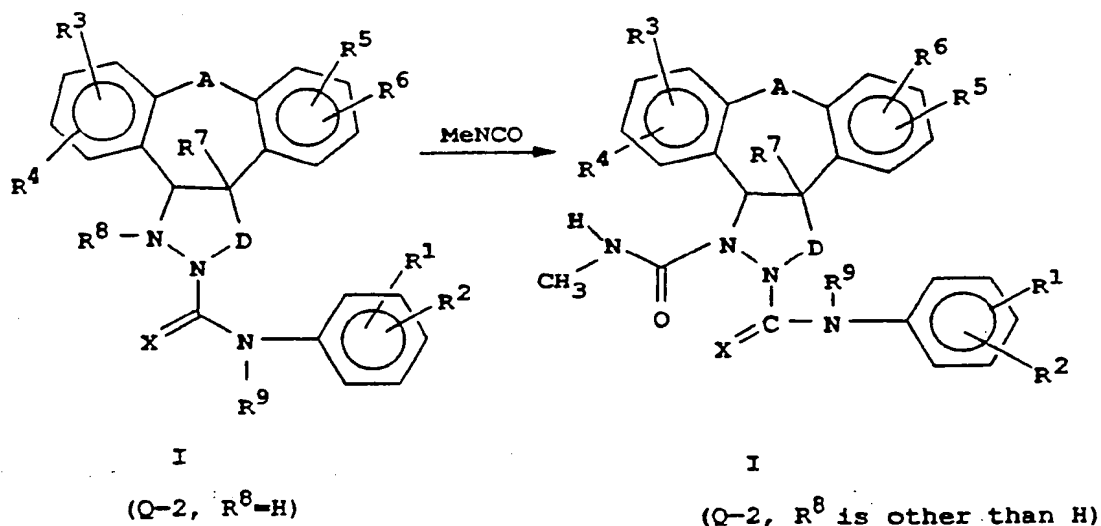
SCHEME 12

5 Compounds of Formula I (Q-2) where R⁸ is not H can be prepared by the reaction of Formula I (Q-2) compounds where R is H with a variety of electrophiles. For example, these electrophiles include, but are not limited to, alkyl halides, alkyl and aryl isocyanates, acyl

10 halides, sulfonyl halides and alkyl chlorocarbonates. Reactions to prepare Formula I (Q-2) compounds where R is not H can be conducted through standard procedures known to those skilled in the art. For example, compounds of Formula I (Q-2) where R⁸ is methylaminocarbonyl, can be

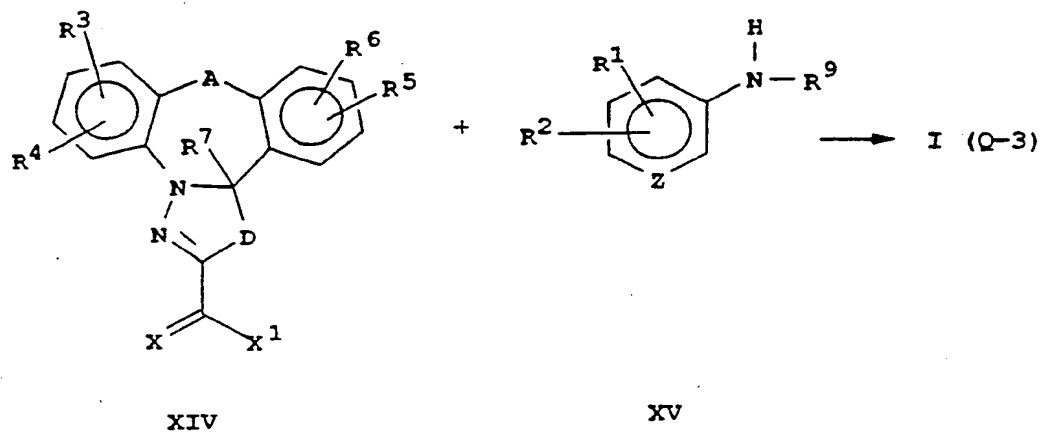
15 prepared by the reaction of Formula I (Q-2) compounds where R⁸ is H with methyl isocyanate (Scheme 13). The reaction can be conducted using equal molar amounts of the reactants in an inert solvent such as ether, tetrahydrofuran, dimethoxyethane, ethyl acetate,

20 methylene chloride and chloroform, and optionally in the presence of a base such as tertiary alkylamines, substituted pyridines, alkali metals, and the like.

SCHEME 13

5 Compounds of Formula I (Q-3) can be prepared by the reaction of activated carbonyl or thiocarbonyl compounds of Formula XIV with substituted anilines or pyridines (XV) in the presence or absence of an acid acceptor or suitable condensing agent. Scheme 14 illustrates this transformation.

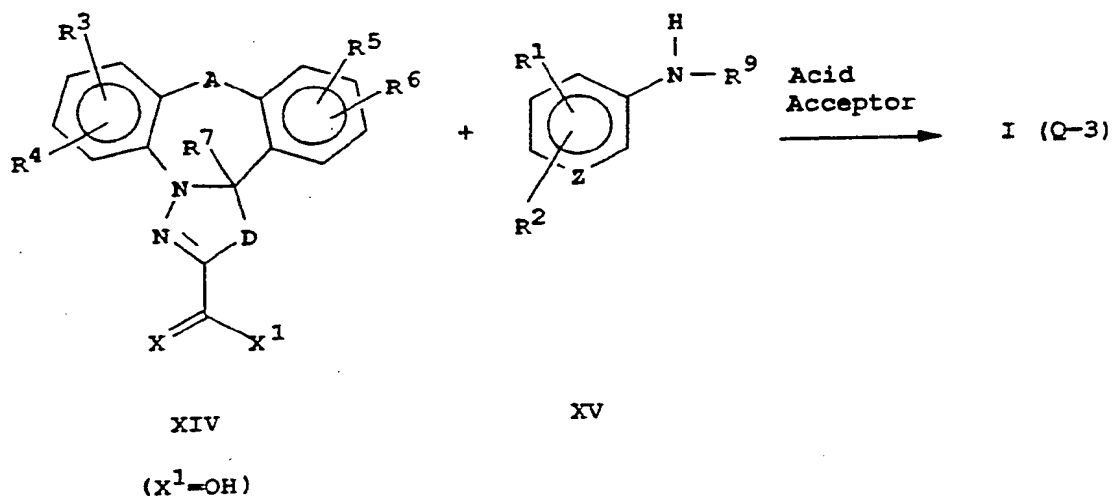
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SCHEME 14

X¹=OH, Cl or C₁ to C₄ alkoxy

One particularly useful method involves the chlorination of an acid derivative (XIV; $X^1 = OH$) with thionyl chloride or another chlorinating agent followed by treatment with XV in the presence of an acid acceptor such as an amine base, preferably triethylamine. Suitable solvents for the chlorination reaction are inert to hydrogen chloride and include benzene, toluene, and dichloromethane. Preferred temperatures for this process are from about 20°C to 100°C with temperatures between about 20°C and 80°C being particularly preferred. The latter reaction can be carried out in many different solvents such as dialkylethers, chlorinated hydrocarbons, and aromatic hydrocarbons. While temperatures at or below 25°C are preferred, higher temperatures can also be employed. These reactions are normally run at atmospheric pressure, but can also be carried out at elevated pressures. Scheme 15 illustrates this transformation.

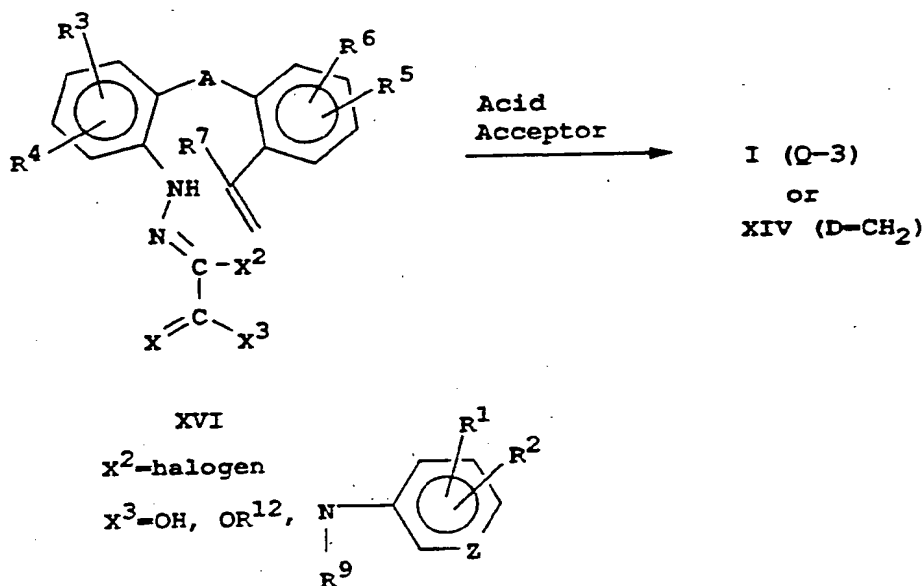
SCHEME 15



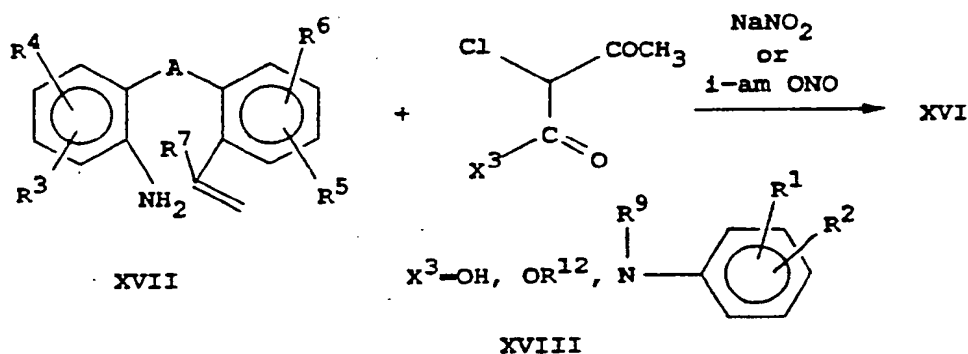
Esters of Formula XIV ($X^1 = C_1$ to C_4 alkoxy) can be converted directly to compounds of Formula I (Q-3) in several ways. In the presence of Lewis acids such as $AlMe_3$, Formula XV compounds react readily with esters of Formula XIV. The reaction is best carried out at room temperature to about $120^\circ C$. Suitable solvents include dichloromethane, 1,2-dichloroethane, and toluene. The method described in *Organic Synthesis* 1979, 59, 49-53, proceeds best with esters of lower alcohols such as methanol or ethanol.

Acids of Formula XIV ($X^1 = OH$) can be converted directly to compounds of Formula I by use of coupling agents known in the peptide art in conjunction with substituted anilines or pyridines. Coupling agents include dicyclohexylcarbodiimide (DCC), N-hydroxy-succinimide, 2-chloro-N-methylpyridinium iodide, carbonyl diimidazole, or other agents capable of activating an acid function or acting as a dehydrating agent.

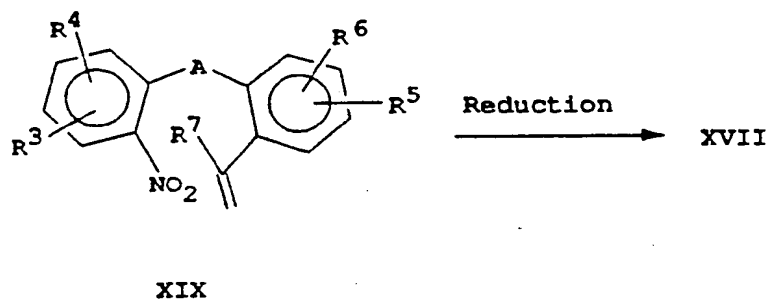
Compounds of Formula I (Q-3, $D = CH_2$) and intermediates of Formula XIV can also be obtained by the intramolecular dipolar cycloaddition reaction of nitrile-imines, generated from substituted phenylhydrazones of Formula XVI (Scheme 16). The presence of an acid acceptor (generally an amine base, for example, triethylamine) is necessary for the formation of the nitrile-imine. Suitable solvents include benzene, toluene, 1,2-dichloroethane, chloroform, and tetrahydrofuran. The reaction can be carried out at temperatures ranging from about $20^\circ C$ to $120^\circ C$ with the relative reactivity of the alkene moiety governing the required temperature for a given example.

SCHEME 16

- 5 The required hydrazones of Formula XVI can be synthesized by the Japp-Klingemann reaction (Scheme 17). The coupling of diazonium salts with active methene compounds is known. The more specific coupling of chloroacetoacetic acid derivatives of Formula XVIII
- 10 containing alkenyl substituents is described in *J. Org. Chem.* **1978**, *43*, 1664-1671 and *J. Org. Chem.*, **1981**, *46*, 1402-1409.

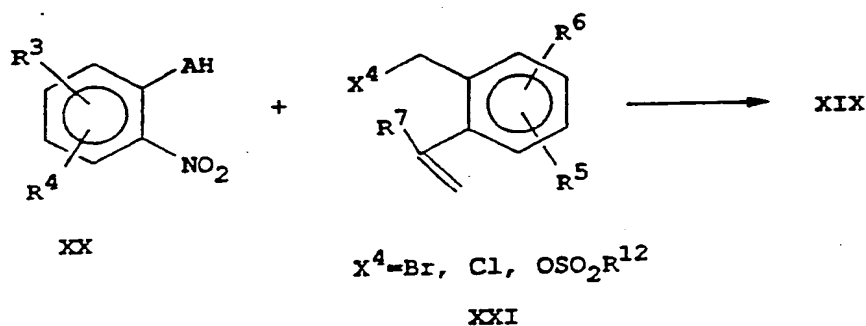
SCHEME 17

- 5 The anilines of Formula XVII can be obtained by the reduction of aromatic nitro compounds of Formula XIX (Scheme 18).. A particularly suitable method involves the treatment of the nitro compound with tin (II) chloride in alcoholic solvents; see *Tetrahedron Letters* 1984, 25, 839-842.

SCHEME 18

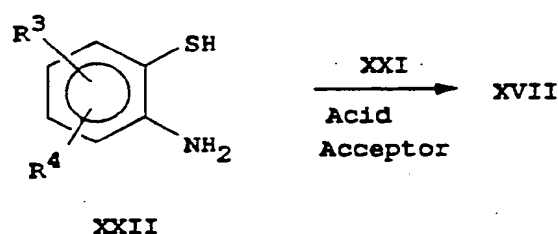
- 15 Nitro compounds of Formula XIX containing a heteroatom in the alkenyl chain can be obtained by alkylation reactions (Scheme 19). Treatment of a substituted phenol, thiophenol, or aniline of Formula XX with an acid acceptor and an allyl or homoallyl halide or sulfonate of Formula XXI gives compounds of Formula XIX as products. Preferred acid acceptors for the process

are inorganic bases such as potassium carbonate. Preferred solvents include dimethylformamide, dimethylsulfoxide, methylethyl ketone, and ethanol. The reaction is generally carried out at room temperature, but higher temperatures may be necessary in some cases.

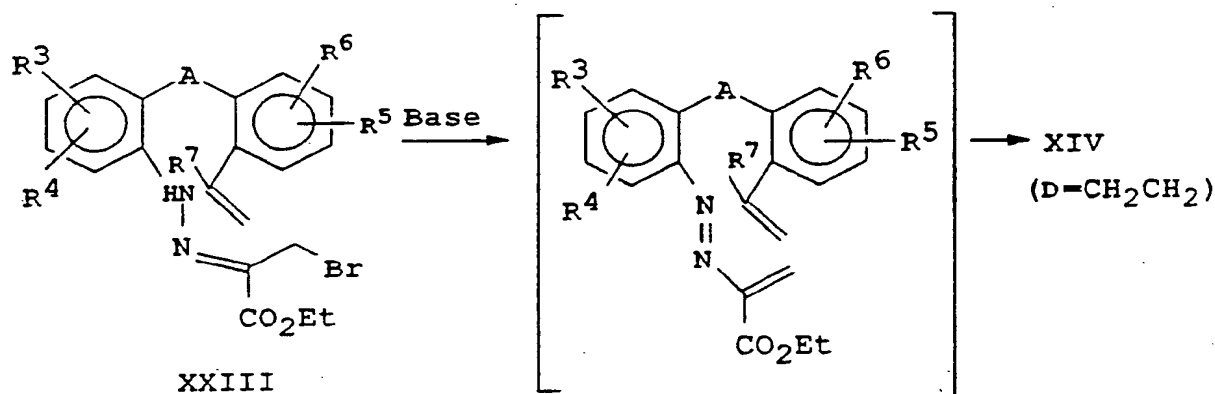
SCHEME 19

10

Anilines of Formula XVII containing sulfur can be obtained by alkylation of the sulfur of Formula XXII (Scheme 20). The conditions which favor this process are alcoholic solvents and sodium hydroxide or sodium alkoxides as bases. The reaction is generally carried out using allylic halides of Formula XXI at about 20°C to 80°C in lower alcoholic solvents, preferably ethanol.

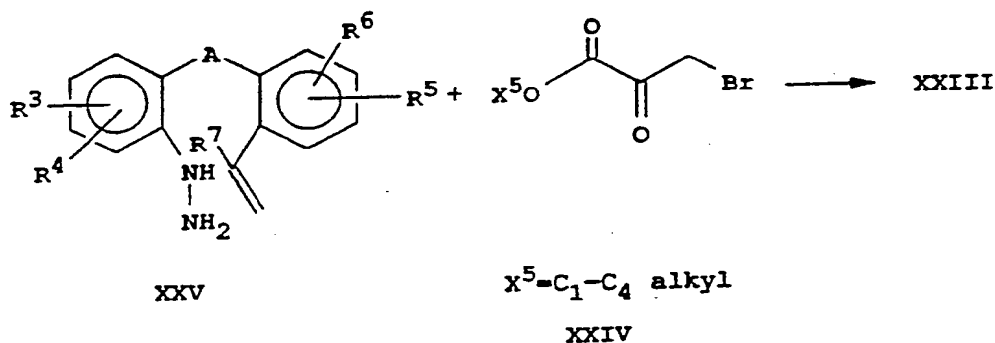
SCHEME 20

- 5 Compounds of Formula XIV, wherein D is CH₂CH₂ can be prepared from Formula XXIII compounds by the reaction of Formula XXIII compounds with a base such as sodium hydride, potassium hydride, sodium carbonate, potassium carbonate, sodium methoxide and lithium diisopropylamide.
- 10 Suitable solvents include methylene chloride, chloroform, tetrahydrofuran, ether and toluene. The reaction temperature can range from 0°C to the reflux temperature of the particular solvent utilized and the reaction is generally complete in 24 hours. Scheme 21 illustrates
- 15 this transformation.

SCHEME 21

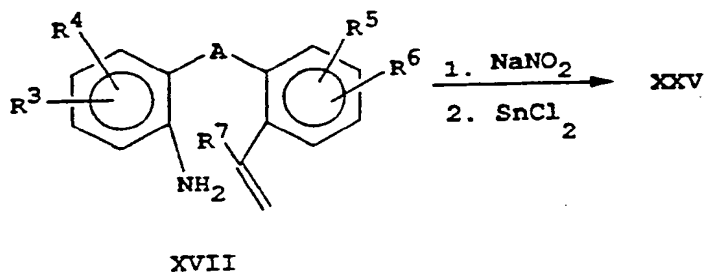
- 20 Compounds of Formula XXIII can be prepared from compounds of the Formula XXV by the reaction with an

equimolar amount of XXIV in conventional organic solvents such as ether, tetrahydrofuran, methanol, ethanol, methylene chloride, benzene and toluene. Typical reaction temperatures can range from room temperature to the reflux temperature of the particular solvent utilized and the reaction is usually complete in 24 hours. Scheme 22 illustrates this transformation.

SCHEME 22

10

Compounds of Formula XXV can be prepared from Formula XVII derivatives by the diazotization/reduction reaction illustrated in Scheme 23.

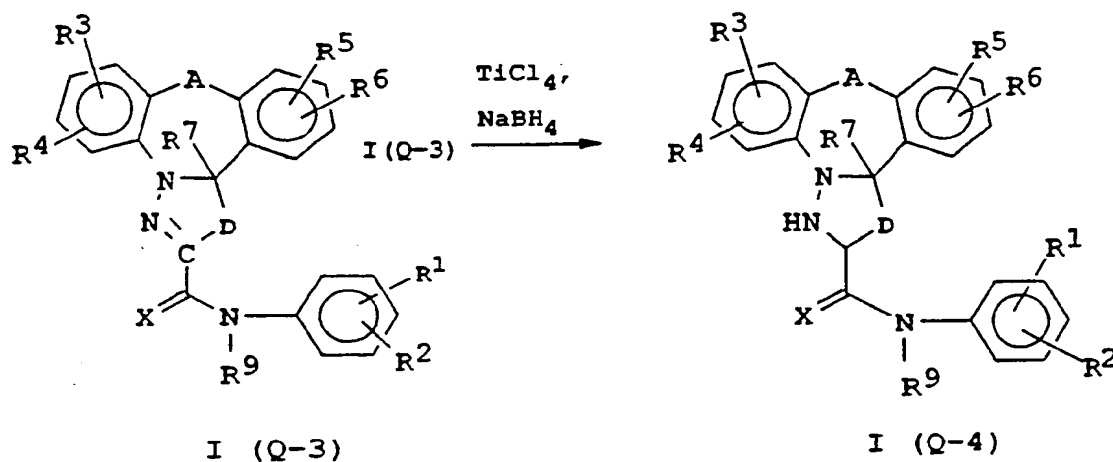
SCHEME 23

15

Compounds of Formula I (Q-4) can be prepared by a titanium tetrachloride/sodium borohydride reduction of Formula I (Q-3) compounds. Scheme 24 illustrates this transformation.

20

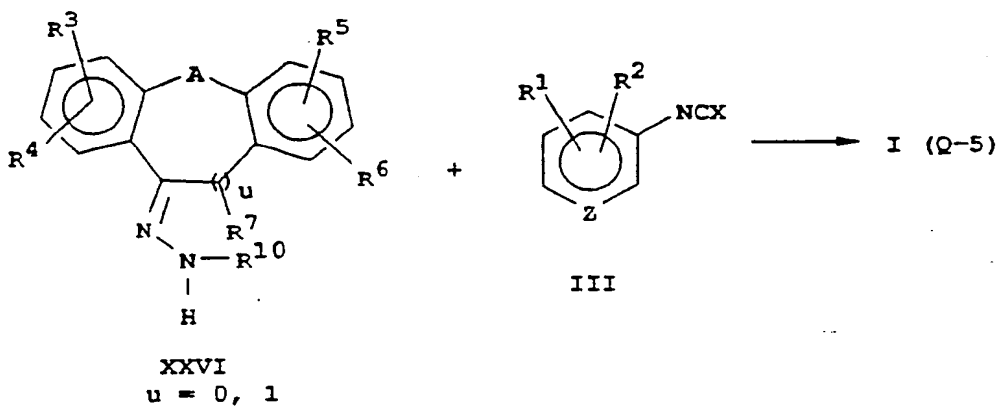
SCHEME 24



5 Compounds of Formula I (Q-4) where R⁸ is not equal to H can be prepared by the reaction of Formula I (Q-4) compounds where R is H with a variety of electrophiles analogous to the procedure described in Scheme 13.

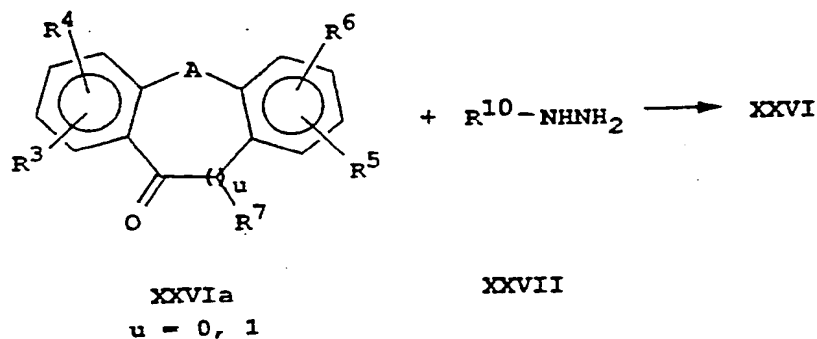
10 Compounds of Formula I (Q-5) can be prepared by the reaction of hydrazones of Formula XXVI with aryl isocyanates of Formula III as shown in Scheme 25.

SCHEME 25



15

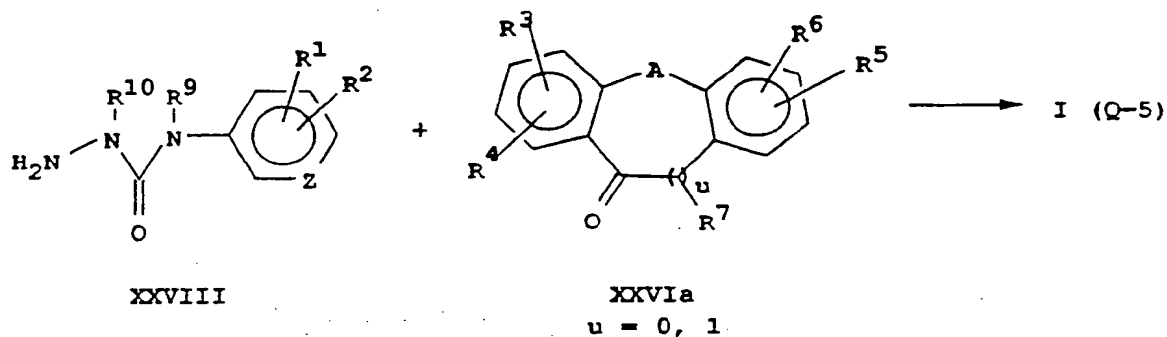
The hydrazones of Formula XXVI can be obtained by processes known in the art involving condensation of a ketone of Formula XXVIa with either hydrazine or a substituted derivative thereof (Formula XXVII). This reaction is typically conducted with equimolar amounts of XXVII and XXVIa although greater than stoichiometric amounts of hydrazine XXVII can be used. Suitable solvents include the alcohols such as methanol, ethanol, propanol, butanol and the like at temperatures in the range of about 0°C to 150°C, with the reflux temperature of the solvent generally being a convenient reaction temperature. Acid catalysts can also be useful, particularly for some of the more sterically hindered Formula XXVIa compounds. Typical acid catalysts include sulfuric, hydrochloric and p-toluene sulfonic acid. Scheme 26 illustrates this transformation.

SCHEME 26

20

An alternate process useful for the preparation of compounds of Formula I (Q-5) involves condensation of a phenyl substituted semicarbazide of Formula XXVIII with a ketone of Formula XXVIa. Preferred conditions for this reaction include an acid catalyst such as hydrochloric, sulfuric, acetic or p-toluene sulfonic acid. Reaction

temperatures can range from about 0°C to 150°C with the reflux temperature of the solvent used generally preferred. Suitable solvents include ethers such as tetrahydrofuran and dioxane; aromatic hydrocarbons such as benzene and toluene. Especially preferred solvents are alcohols such as methanol, ethanol and isopropanol. Scheme 27 illustrates this transformation.

SCHEME 27

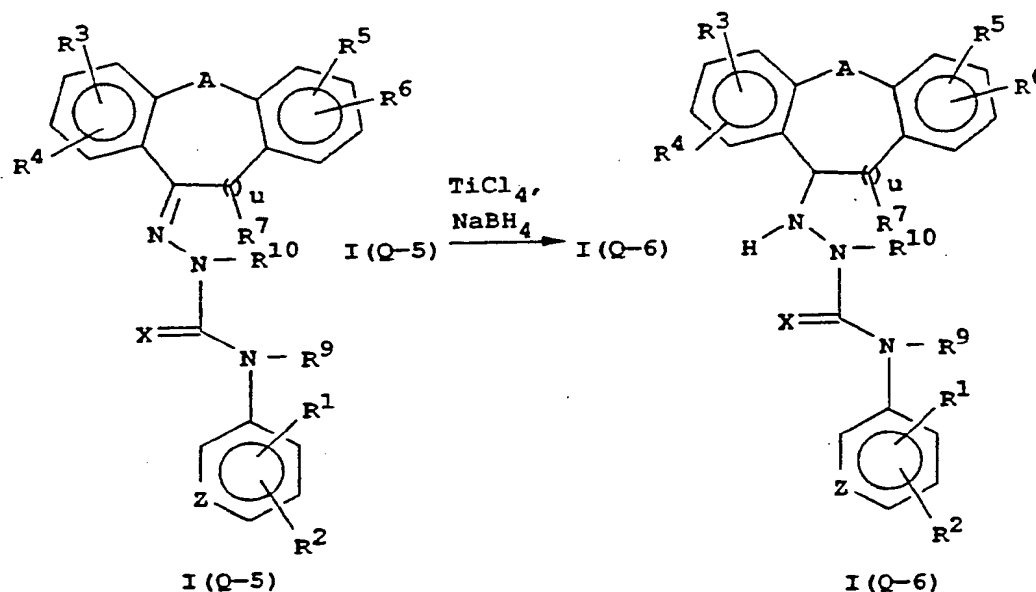
10

Compounds of Formula I (Q-5) where R¹⁰ and R⁹ are other than H can be prepared from the corresponding compounds where R¹⁰ and R⁹ are H by reaction with electrophilic reagents such as alkyl halides, acyl halides, alkyl chloroformates and sulfenyl halides. The use of a base is generally preferred in these reactions but is dependent upon the specific nature of the reactants. For example, when the electrophilic reagent is selected from an alkyl halide, acyl halide or alkyl chloroformate, then metal hydrides such as sodium hydride or potassium hydride in solvents such as tetrahydrofuran or dimethylformamide are preferred. When sulfenyl halides are used, then amine bases such as triethylamine in solvents such as diethyl ether or tetrahydrofuran are generally preferred. Of course, many of the compounds where R¹⁰ is other than H can also be prepared by use of

the appropriate hydrazine XXVII as in Scheme 26. For example, methyl hydrazine and methyl carbazate will produce compounds where R^{10} is methyl and carbomethoxy, respectively. Compounds of Formula XXVIa where $u = 0$ are
 5 recognized to be dibenzosuberones, benzoxapines, etc. of which preparations are documented in the literature (EP-68,370, Monat. Fur. Chemie 1962, 93, 889).

Compounds of Formula I (Q-6) can be prepared by a titanium tetrachloride/sodium borohydride reduction of
 10 Formula I (Q-5) compounds. Scheme 28 illustrates this transformation.

SCHEME 28



15

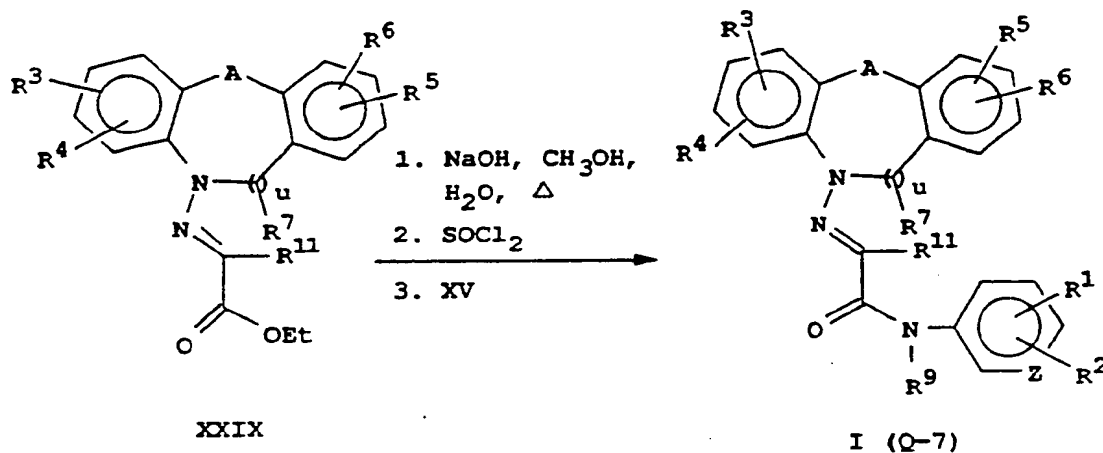
Compounds of Formula I (Q-6) where R^8 is not H can be prepared by the reaction of Formula I (Q-5) compounds where R is H with a variety of electrophiles analogous to the procedure described in Scheme 13.

20

Compounds of Formula I (Q-7) can be prepared whereby Formula XXIX esters are saponified, converted to the acid chloride and reacted with an appropriately substituted

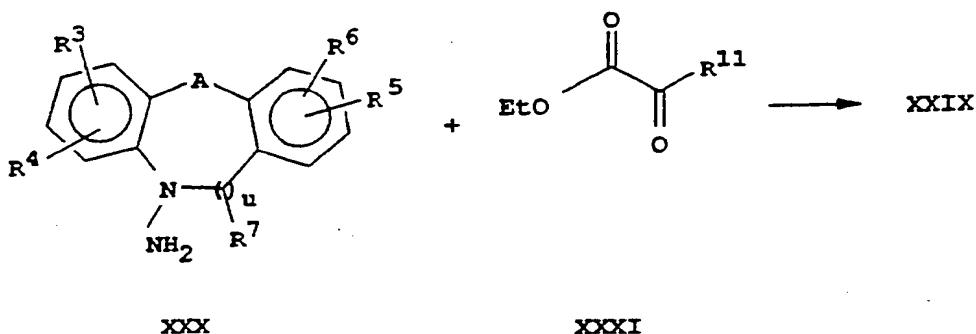
aniline or pyridine. Scheme 29 illustrates this transformation.

SCHEME 29



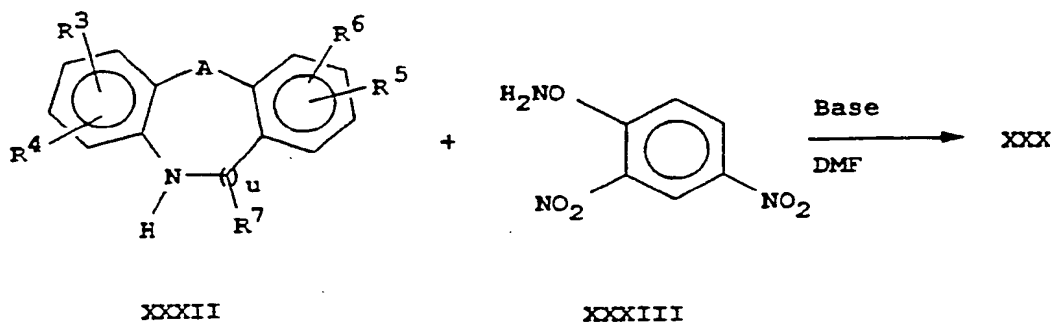
5

Formula XXIX compounds can be prepared by the reaction of Formula XXX hydrazines with well known esters of Formula XXXI. The reaction can be conducted in the presence or absence of an acid or base in an unreactive solvent system such as methanol, ethanol, methylene chloride, chloroform, tetrahydrofuran and dioxane, but not limited to these. The temperature of the reaction can be varied from 0°C to the reflux temperature of the particular solvent. The reaction is usually complete in 24 hours. Scheme 30 illustrates this transformation.

SCHEME 30

5 Compounds of Formula XXX can be prepared by the reaction of Formula XXXII derivatives with the reagent O-(2,4-dinitrophenyl)hydroxylamine (XXXIII) in the presence of a base such as sodium carbonate, sodium bicarbonate or potassium carbonate in a nonreactive solvent such as dimethylformamide, dimethylsulfoxide, tetrahydrofuran and dioxane. The reaction temperature can vary from 0°C to 100°C with 25°C being preferred. The reaction is usually complete in 24 hours. Scheme 31 illustrates this transformation.

15

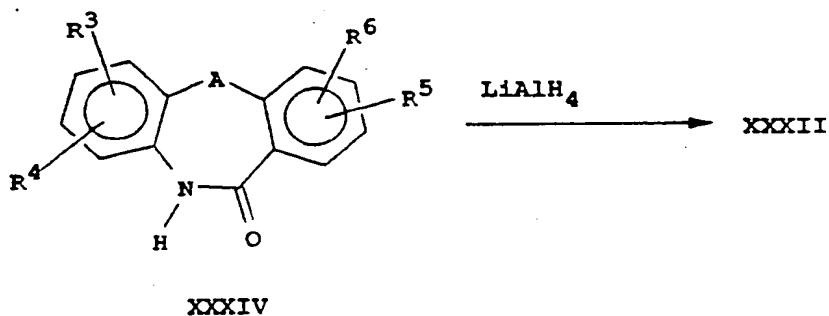
SCHEME 31

20 Compounds of Formula XXXII (u = 0) are known in the literature (Annalen der Chem. 1969, 723, 95; J.Het. Chem.

1981, 18, 855) and can be prepared by one skilled in the art by obvious modifications therein.

Compounds of Formula XXXII ($u = 1$) can be prepared by the reduction of Formula XXXIV compounds shown in
 5 Scheme 32, using reagents such as LiAlH_4 or BH_3 in a similar fashion as described in Scheme 4.

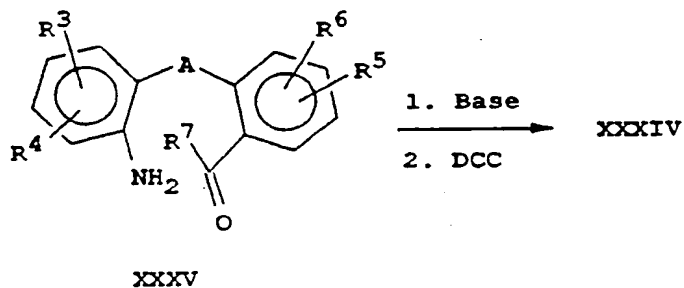
SCHEME 32



10

Compounds of Formula XXXIV can be prepared by the reaction of Formula XXXV in the presence or absence of a base with a coupling reagent such as dicyclohexyl-carbodiimide (DCC). Scheme 33 illustrates this
 15 transformation.

SCHEME 33

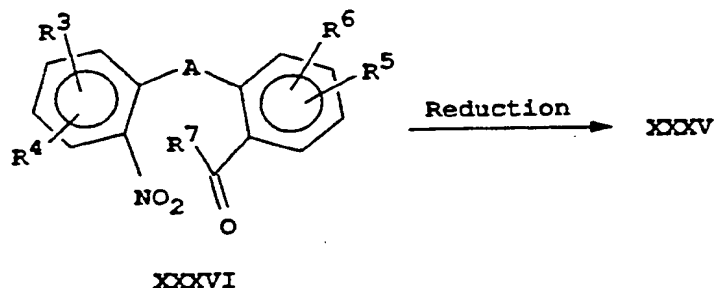


20

The anilines of Formula XXXV can be obtained by the reduction of aromatic nitro compounds of Formula XXXVI

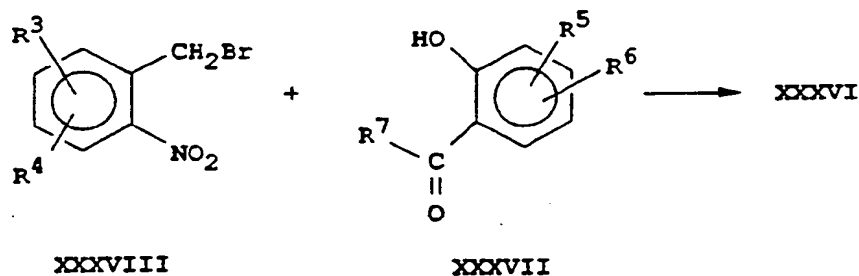
analogous to the reaction described in Scheme 18. Scheme 34 illustrates this transformation.

SCHEME 34



5

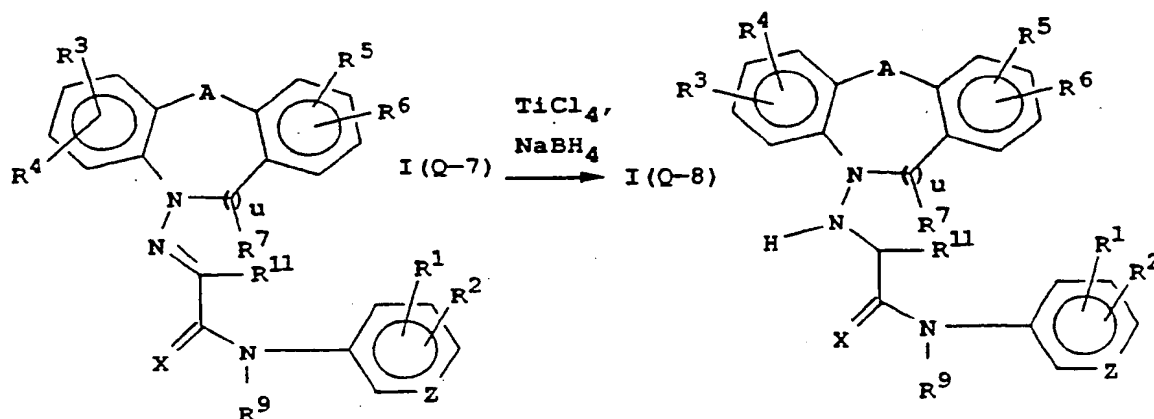
Compounds of Formula XXXVI can be prepared by reaction of alcohols of Formula XXXVII with activated halides of Formula XXXVIII. This type of reaction is known in the art and is carried out in the presence of a suitable base such as sodium hydride, potassium tert-butoxide, or other strong bases. This sequence can be carried out in a variety of solvents provided they do not react with the base. Examples of such solvents include tetrahydrofuran, dimethylformamide, dioxane, or mixtures thereof. Alternatively, the coupling can be carried out under phase-transfer conditions using an alkali hydroxide as base and a tetraalkylammonium salt as catalyst. The halides of Formula XXXVIII are commercially available or can be readily synthesized by those skilled in the art. Scheme 35 illustrates this transformation.

SCHEME 35

5 Compounds of Formula I (Q-8) can be prepared by a titanium tetrachloride/sodium borohydride reduction of Formula I (Q-7) compounds. Scheme 36 illustrates this transformation.

SCHEME 36

10



15 Compounds of Formula I (Q-8) where R⁸ is not H can be prepared by the reaction of Formula I (Q-7) compounds where R⁸ is H with a variety of electrophiles analogous to the procedure described in Scheme 13.

The following Examples serve to further illustrate the invention.

EXAMPLE 12-(10,11-Dihydro-5H-dibenzo[A,D]-cyclohepten-5-ylidene)-
N-[4-(trifluoromethoxy)phenyl]hydrazinecarboxamide

To a solution of 1.0 g (4.8 mmol) of dibenzosuberone
5 dissolved in 40 mL of n-butanol was added 0.77 g (24.0
mmol) of hydrazine and 1.5 g (24.0 mmol) of acetic acid.
The reaction mixture was refluxed for 48 h, cooled and
concentrated under reduced pressure. Then, 100 mL of
saturated NaHCO₃ solution was added and was extracted
10 with ethyl acetate (3 x 50 mL). The organic phase was
washed with brine (1 x 100 mL), dried over anhydrous
magnesium sulfate and filtered into a flask to which was
added 0.97 g (4.8 mmol) of 4-trifluoromethoxy-phenyl
isocyanate and the reaction mixture was stirred at room
15 temperature for 30 minutes. The reaction mixture was
concentrated under reduced pressure and flash-
chromatographed using 20% ethyl acetate/80% hexanes as
eluent to afford 0.10 g of a white solid, mp 172-173°C.
1H NMR (CDCl₃) δ 2.8-3.4 (m, 4H), 7.1-7.7 (m, 12H), 8.0
20 (bs, 1H), 8.38 (bs, 1H).
IR (mineral oil) 3250, 1667 cm⁻¹.

EXAMPLE 2

Part A: 2-[(2-Bromophenyl)methoxy]-3-fluorobenzonitrile
25 To 10.9 g (79.1 mmol) of potassium carbonate
suspended in 100 mL DMF was added 10.0 g (71.9 mmol) 2,3-
difluorobenzonitrile and 14.8 g (79.1 mmol)
2-bromobenzyl alcohol. The reaction mixture was heated
at 70°C for 18 h, cooled and poured into 250 mL 10%
30 HCl/ice and extracted with ethyl ether (3 x 150 mL). The
ether layer was washed with water (1 x 100 mL) and brine
(1 x 100 mL), dried over anhydrous magnesium sulfate,
filtered and concentrated under reduced pressure to
afford 12.8 g of a white solid.

^1H NMR (CDCl_3) δ 5.41 (s, 2H), 7.1-7.4 (m, 5H), 7.58 (d, 1H), 7.7 (d, 1H). IR (mineral oil) 2236 cm^{-1} .

Part B: 4-Fluorodibenz[B,E]oxepin-11(6H)-one

5 To a cooled (-78°C) solution of 5.0 g (16.3 mmol) of the product from Part A dissolved in 150 mL THF was added dropwise over 10 minutes 7.2 mL (17.9 mmol) of a 2.5 M solution of n-butyllithium in hexanes. The reaction mixture was stirred at -78°C for 15 minutes and then
10 allowed to warm to room temperature where 150 mL 10% HCl was added and stirred for 18 h. A white solid precipitate was filtered and dissolved in ether (100 mL), dried over anhydrous magnesium sulfate, filtered and concentrated under reduced pressure to afford 2.6 g of a
15 white solid, m.p. $108-110^\circ\text{C}$.

^1H NMR (CDCl_3) δ 5.29 (s, 2H), 7.02 (m, 1H), 7.3-7.6 (m, 4H), 7.9-8.02 (m, 2H). IR (mineral oil) 1642 cm^{-1} .

Part C:

20 2-(4-Fluorodibenz[B,E]oxepin-11-(6H)-ylidene)-
N-[4-trifluoromethoxy]phenyl]hydrazine carboxamide

To a solution of 75 mL n-butanol was added 2.38 g (10.4 mmol) of the product from Part B and 1.67 g (52.2 mmol) hydrazine (anhydrous) and 3.13 g (52.2 mmol) acetic
25 acid. The reaction mixture was heated at reflux for 72 h, concentrated under reduced pressure and 100 mL ethyl acetate was added. The ethyl acetate layer was extracted with water (2 x 100 mL), saturated sodium bicarbonate (1 x 100 mL) and brine (1 x 100 mL) and dried over anhydrous
30 magnesium sulfate, filtered and concentrated to afford a viscous oil. The crude product was flash-chromatographed using 10% ethyl acetate/90% hexane as eluent and afforded 0.34 g of a white solid which was added 0.31 g (1.4 mmol)

4-trifluoromethoxyphenylisocyanate and 0.21 (1.5 mmol) triethylamine. The reaction mixture was stirred at room temperature overnight. The reaction mixture was concentrated and triturated with ethyl ether and filtered to afford 0.38 g of a white solid, m.p. 188-191°C. ¹H NMR (CDCl₃) δ 5.08 (m, 1H), 5.4 (m, 1H), 6.95 (m, 1H), 7.12 (dt, 1H), 7.20 (d, 2H), 7.4-7.6 (m, 7H), 8.23 (s, 1H), 8.38 (s, 1H). IR (mineral oil) 3370, 3100, 1688 cm⁻¹.

EXAMPLE 3

3,3a,9,10-Tetrahydro-N-[4-(trifluoromethyl)phenyl]-2H-dibenzo[3,4:7,8]cycloocta[1,2-c]pyrazole-2-carboxamide

To a suspension of dibenzocyclooctanone (1.0 g, 4.5 mmol) in 12 mL methanol was added 1.4 mL 37% aqueous formaldehyde (18.5 mmol), 50 µL pyridine (0.7 mmol), and 50 µL glacial acetic acid (0.8 mmol). The reaction mixture was heated at reflux for 100 hr, cooled, and concentrated under reduced pressure. Then, 20 mL water was added and extracted with dichloromethane (2 x 30 mL). The organic phase was dried over anhydrous MgSO₄, filtered and concentrated in vacuo.

The crude product was dissolved in 30 mL absolute ethanol and 0.3 mL hydrazine hydrate (6.0 mmol) was added. The reaction mixture was heated at reflux for 1 hr, cooled, and concentrated under reduced pressure. Then, 20 mL of H₂O was added and was extracted with ether (2 x 50 mL). The organic phase was dried over anhydrous magnesium sulfate, divided into two equal portions, and filtered into a flask containing 0.32 mL of α,α,α-trifluoro-p-tolyl isocyanate (2.25 mmol). After 4 hr, the solvent was removed under reduced pressure to give an oily product which was triturated with hexane and

ethylacetate, and filtered to afford 0.067 g of a yellow solid, m.p. 245-250°C.

^1H NMR (CDCl_3) δ 8.24 (1H, bs); 7.71-7.22 (4H, m), 7.19-7.05 (8H, m); 5.15 (1H, dd, $J=10$ Hz, 12 Hz);

5 4.62-4.36 (2H, m); 3.41-3.21 (4H, m), m.p. 245-250°C.

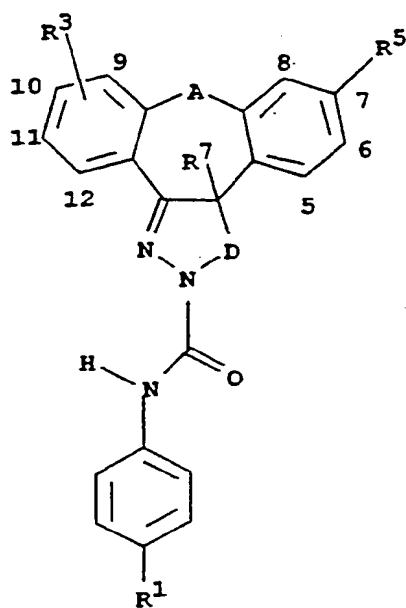
By the general procedures described herein, or obvious modifications thereof, the compounds of Tables 1 to 9 can be prepared. The Tables are arranged in a format such that maximum utilization of space is
10 achieved. An interpretation of the first example of Table 1 is provided. The variables D, R^1 , R^3 , R^5 and R^7 are set as described and the variable A can retain 7 separate descriptors, the results of which are the description of 7 distinct compounds.

15

20

25

30

TABLE 1

D is CH₂
 R¹ is Cl
 R³ is H
 R⁵ is H
 R⁷ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂
 O
 S

D is CH₂
 R¹ is Cl
 R³ is 9-F
 R⁵ is H
 R⁷ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂
 O
 S

D is CH₂
 R¹ is Cl
 R³ is 10-F
 R⁵ is H
 R⁷ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂
 O
 S

D is CH₂
R¹ is Cl
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is H
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 9-F
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-F
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is H
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-Br
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 9-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 9-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 10-Br
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is Cl
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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S

D is CH₂
R¹ is Cl
R³ is 9-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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D is CH₂
R¹ is Cl
R³ is 10-F
R⁵ is H
R⁷ is Me

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CH₂CH₂
OCH₂
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N(Me)CH₂
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D is CH₂
R¹ is Cl
R³ is 10-Cl
R⁵ is H
R⁷ is Me

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CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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D is CH₂
R¹ is Cl
R³ is H
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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D is CH₂
R¹ is Cl
R³ is 10-Br
R⁵ is H
R⁷ is Me

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CH₂CH₂
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D is CH₂
R¹ is Cl
R³ is 9-F
R⁵ is F
R⁷ is Me

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R¹ is Cl
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R¹ is Cl
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R¹ is Cl
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R⁵ is H
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R¹ is Br
R³ is 9-Cl
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D is CH₂
R¹ is Br
R³ is 10-F
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R⁷ is Me

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D is CH₂
R¹ is Br
R³ is 10-Cl
R⁵ is F
R⁷ is Me

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CH₂CH₂
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NHCH₂
N(Me)CH₂
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D is CH₂
R¹ is CF₃
R³ is H
R⁵ is H
R⁷ is H

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CH₂CH₂
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D is CH₂
R¹ is Br
R³ is 10-Br
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R¹ is CF₃
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NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₂H
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₂H
R³ is 10-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₂H
R³ is 10-Cl
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₃
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₂H
R³ is 10-Br
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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D is CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is H
R⁷ is H

A

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R¹ is OCF₂H
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R¹ is OCF₃
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R⁵ is H
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R¹ is OCF₃
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R¹ is OCF₃
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R¹ is OCF₃
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R¹ is OSO₂CF₃
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R¹ is OSO₂CF₃
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R¹ is Cl
R³ is H
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A

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S

D is CH₂CH₂
R¹ is Br
R³ is 10-Br
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is Br
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is Br
R³ is 9-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is Br
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is Br
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is Br
R³ is H
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
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S

D is CH₂CH₂
R¹ is Br
R³ is 10-Br
R⁵ is H
R⁷ is H

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CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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S

D is CH₂CH₂
R¹ is Br
R³ is 9-F
R⁵ is F
R⁷ is H

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CH₂CH₂
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NHCH₂
N(Me)CH₂
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S

D is CH₂CH₂
R¹ is Br
R³ is 9-Cl
R⁵ is H
R⁷ is H

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CH₂CH₂
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N(Me)CH₂
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D is CH₂CH₂
R¹ is Br
R³ is 10-F
R⁵ is F
R⁷ is H

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CH₂CH₂
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D is CH₂CH₂
R¹ is Br
R³ is 10-Cl
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CH₂CH₂
OCH₂
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NHCH₂
N(Me)CH₂
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S

D is CH₂CH₂
R¹ is Br
R³ is H
R⁵ is Cl
R⁷ is H

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CH₂CH₂
OCH₂
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N(Me)CH₂
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S

D is CH₂CH₂
R¹ is Br
R³ is 10-Br
R⁵ is F
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R¹ is Br
R³ is 9-F
R⁵ is Cl
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R¹ is Br
R³ is 10-F
R⁵ is Cl
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R¹ is Br
R³ is 10-Cl
R⁵ is Cl
R⁷ is H

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OCH₂
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NHCH₂
N(Me)CH₂
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D is CH₂CH₂
R¹ is Br
R³ is H
R⁵ is H
R⁷ is Me

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CH₂CH₂
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R¹ is Br
R³ is 10-Br
R⁵ is Cl
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R¹ is Br
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D is CH₂CH₂
R¹ is CF₃
R³ is H
R⁵ is H
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R¹ is CF₃
R³ is H
R⁵ is H
R⁷ is Me

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R¹ is OCF₂H
R³ is H
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R¹ is CF₃
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R¹ is OCF₂H
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SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is H
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Br
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Br
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is H
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Br
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-Cl
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is H
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OCF₃
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is H
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is F
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is Cl
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is H
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂CH₂
R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is F
R⁷ is Me

A

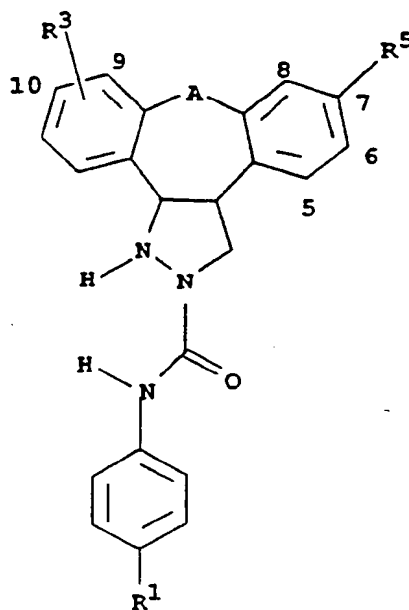
CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is F
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

TABLE 2

R¹ is CF₃R³ is 9-FR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂R¹ is OCF₃R³ is 9-FR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂R¹ is CF₃R³ is 9-ClR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂R¹ is OCF₃R³ is 9-ClR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂R¹ is CF₃R³ is 10-ClR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂R¹ is OCF₃R³ is 10-ClR⁵ is H

A

CH₂CH₂OCH₂SCH₂NHCH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is F

A

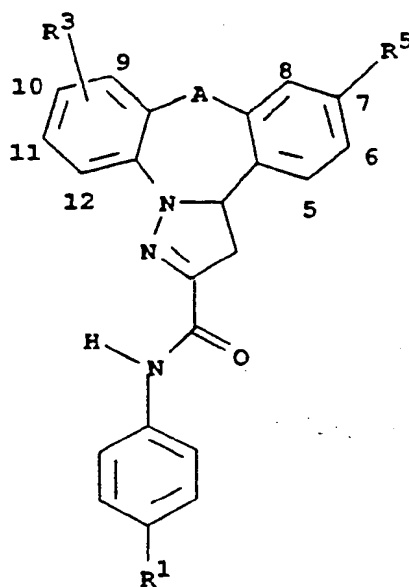
CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

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TABLE 3

R¹ is CF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is F

A

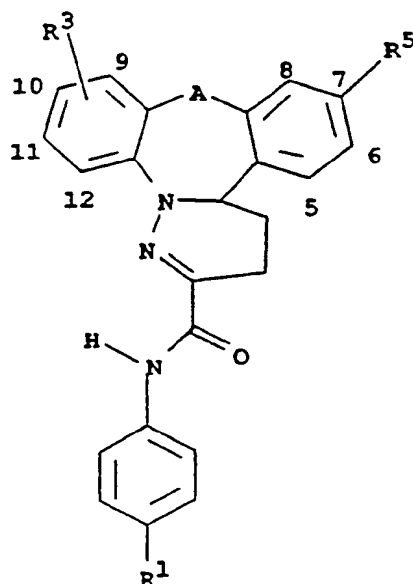
CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

TABLE 4



R¹ is CF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is F

A

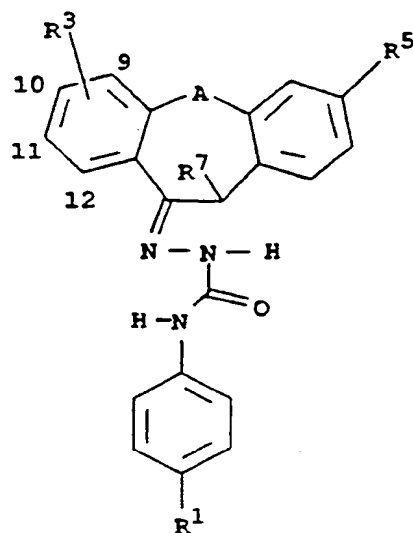
CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

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TABLE 5

R^1 is Cl
 R^3 is H
 R^5 is H
 R^7 is H

A

CH_2CH_2
 OCH_2
 SCH_2
 NHCH_2
 $\text{N}(\text{Me})\text{CH}_2$
 O
 S

R^1 is Cl
 R^3 is 9-F
 R^5 is H
 R^7 is H

A

CH_2CH_2
 OCH_2
 SCH_2
 NHCH_2
 $\text{N}(\text{Me})\text{CH}_2$
 O
 S

R^1 is Cl
 R^3 is 10-F
 R^5 is H
 R^7 is H

A

CH_2CH_2
 OCH_2
 SCH_2
 NHCH_2
 $\text{N}(\text{Me})\text{CH}_2$
 O
 S

R¹ is Cl
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Cl
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is Br
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is CF₃
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₃
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₂H

R³ is H

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 9-Cl

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is H

R⁵ is H

R⁷ is Me

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 9-F

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 10-Cl

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 9-F

R⁵ is H

R⁷ is Me

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 10-F

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 10-Br

R⁵ is H

R⁷ is H

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H

R³ is 10-F

R⁵ is H

R⁷ is Me

A

CH₂CH₂

OCH₂

SCH₂

NHCH₂

N(Me)CH₂

O

S

R¹ is OCF₂H
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is H
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₂H
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OCF₂H
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is H
R⁷ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

R¹ is OSO₂CF₃
R³ is H
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

R¹ is OSO₂CF₃
R³ is 9-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

R¹ is OSO₂CF₃
R³ is 9-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

R¹ is OSO₂CF₃
R³ is 10-Cl
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

R¹ is OSO₂CF₃
R³ is 10-F
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

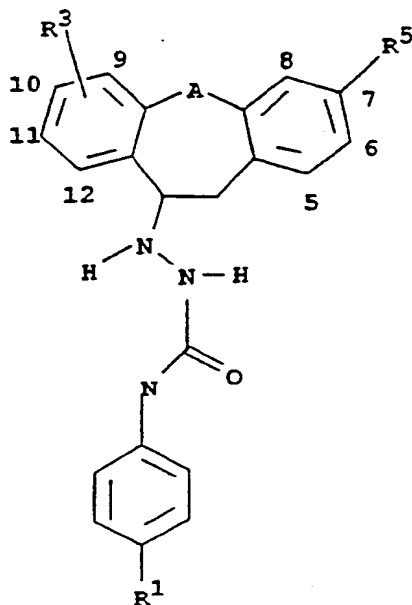
R¹ is OSO₂CF₃
R³ is 10-Br
R⁵ is H
R⁷ is Me

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂
O
S

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TABLE 6



R¹ is CF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

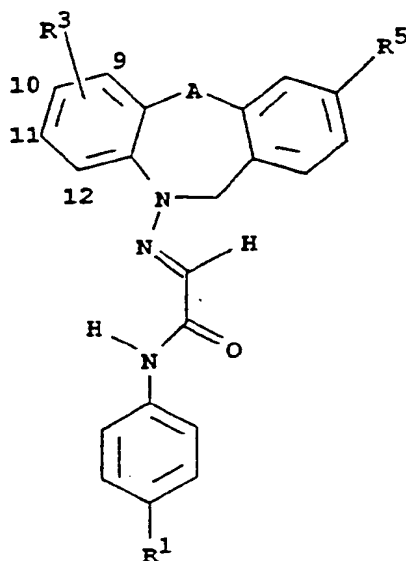
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

118

TABLE 7



R¹ is CF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 9-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is OCF₃
 R³ is 10-Cl
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

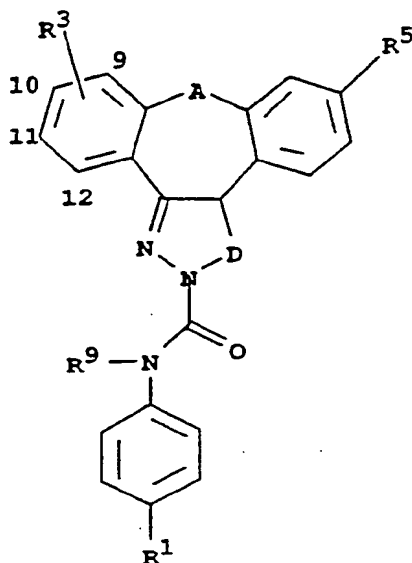
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂

120

TABLE 8



D is CH₂
 R¹ is CF₃
 R³ is H
 R⁵ is F
 R⁹ is Me
 Z is CH
 X is O

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

D is CH₂
 R¹ is CF₃
 R³ is H
 R⁵ is F
 R⁹ is MeCO
 Z is CH
 X is O

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

D is CH₂
 R¹ is CF₃
 R³ is H
 R⁵ is F
 R⁹ is MeO₂C
 Z is CH
 X is O

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is Cl
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is F
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is Cl
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is Cl
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is H
R⁵ is Cl
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is H
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is H
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is H
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9F
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9F
R⁵ is F
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9-F
R⁵ is F
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 9F
R⁵ is F
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is F
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is F
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is F
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-F
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is F
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is F
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is H
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is CF₃
R³ is 10-Cl
R⁵ is F
R⁹ is H
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CH₂CH₂
OCH₂
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NHCH₂
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R³ is H
R⁵ is Cl
R⁹ is Me
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R⁹ is H
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OCH₂
SCH₂
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R⁵ is Cl
R⁹ is H
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OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is H
R⁹ is MeO₂C
Z is CH
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
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NHCH₂
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R¹ is OCF₃
R³ is 10-Cl
R⁵ is H
R⁹ is H
Z is CH
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 9-F
R⁵ is F
R⁹ is H
Z is CH
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

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R¹ is OCF₃
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R⁹ is MeCO
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SCH₂
NHCH₂
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NHCH₂
N(Me)CH₂

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R¹ is OCF₃
R³ is 9-F
R⁵ is F
R⁹ is MeO₂C
Z is CH
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OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁹ is MeCO
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁹ is H
Z is N
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁹ is MeO₂C
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁹ is Me
Z is CH
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-F
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁹ is MeCO
Z is CH
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

D is CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁹ is MeO₂C
Z is CH
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A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

D is CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁹ is H
Z is CH
X is S

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

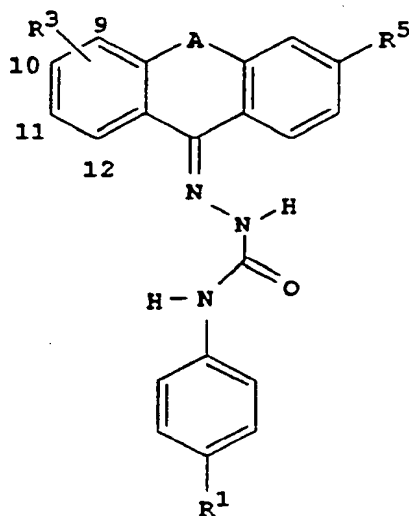
D is CH₂
R¹ is OCF₃
R³ is 10-Cl
R⁵ is F
R⁹ is H
Z is N
X is O

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂
O
S

135

TABLE 9



R¹ is CF₃
 R³ is H
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

R¹ is CF₃
 R³ is 9-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

R¹ is CF₃
 R³ is 10-F
 R⁵ is H

A

CH₂CH₂
 OCH₂
 SCH₂
 NHCH₂
 N (Me) CH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-Br
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 10-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-Br
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is H
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-F
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 10-F
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is CF₃
R³ is 9-Cl
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is H
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is CF₃
R³ is 9-Br
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is CF₃
R³ is 10-Cl
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 10-F
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-Br
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 10-F
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 10-Cl
R⁵ is H

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N(Me)CH₂

R¹ is OCF₃
R³ is 9-Br
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is OCF₃
R³ is H
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is OCF₃
R³ is 10-Cl
R⁵ is F

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is OCF₃
R³ is 9-F
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R¹ is OCF₃
R³ is 10-F
R⁵ is Cl

A

CH₂CH₂
OCH₂
SCH₂
NHCH₂
N (Me) CH₂

R^1 is OCF_3 R^3 is 9-Cl R^5 is Cl

A

 CH_2CH_2 OCH_2 SCH_2 $NHCH_2$ $N(Me)CH_2$ R^1 is OCF_3 R^3 is 9-Br R^5 is Cl

A

 CH_2CH_2 OCH_2 SCH_2 $NHCH_2$ $N(Me)CH_2$ R^1 is OCF_3 R^3 is 10-Cl R^5 is Cl

A

 CH_2CH_2 OCH_2 SCH_2 $NHCH_2$ $N(Me)CH_2$

Formulation and Use

The compounds of this invention will generally be used in formulation with an agriculturally suitable carrier comprising a liquid or solid diluent or an organic solvent. Useful formulations of the compounds of Formula I can be prepared in conventional ways. They include dusts, granules, baits, pellets, solutions, suspensions, emulsions, wettable powders, emulsifiable concentrates, dry flowables and the like. Many of these can be applied directly. Sprayable formulations can be extended in suitable media and used at spray volumes of from about one to several hundred liters per hectare. High strength compositions are primarily used as intermediates for further formulation. The formulations, broadly, contain from less than about 1% to 99% by weight of active ingredient(s) and at least one of a) about 0.1% to 20% surfactant(s) and b) about 5% to 99% solid or liquid diluent(s). More specifically, they will contain effective amounts of these ingredients in the following approximate proportions:

| | | Percent by Weight | | |
|----|---|----------------------|------------|---------------|
| | | Active Ingredient | Diluent(s) | Surfactant(s) |
| 25 | Wettable Powders | 25-90 | 0-74 | 1-10 |
| | Oil Suspensions, Emulsions, Solutions, (including Emulsifiable Concentrates) | 5-50 | 40-95 | 0-15 |
| 30 | Dusts | 1-25 | 70-99 | 0-5 |
| | Granules, Baits and Pellets | 0.01-95 | 5-99 | 0-15 |
| 35 | High Strength Compositions | 90-99 | 0-10 | 0-2 |

Lower or higher levels of active ingredient can, of course, be present depending on the intended use and the physical properties of the compound. Higher ratios of surfactant to active ingredient are sometimes desirable, and are achieved by incorporation into the formulation or by tank mixing.

Typical solid diluents are described in Watkins, et al., "Handbook of Insecticide Dust Diluents and Carriers", 2nd Ed., Dorland Books, Caldwell, New Jersey. The more absorptive diluents are preferred for wettable powders and the denser ones for dusts. Typical liquid diluents and solvents are described in Marsden, "Solvents Guide," 2nd Ed., Interscience, New York, 1950. Solubility under 0.1% is preferred for suspension concentrates; solution concentrates are preferably stable against phase separation at 0°C. "McCutcheon's Detergents and Emulsifiers Annual", Allured Publ. Corp., Ridgewood, New Jersey, as well as Sisely and Wood, "Encyclopedia of Surface Active Agents", Chemical Publ. Co., Inc., New York, 1964, list surfactants and recommended uses. All formulations can contain minor amounts of additives to reduce foam, caking, corrosion, microbiological growth, etc. Preferably, ingredients should be approved by the U.S. Environmental Protection Agency for the use intended.

The methods of making such compositions are well known. Solutions are prepared by simply mixing the ingredients. Fine solid compositions are made by blending and, usually, grinding as in a hammer or fluid energy mill. Suspensions are prepared by wet milling (see, for example, U.S. 3,060,084). Granules and pellets can be made by spraying the active material upon preformed granular carriers or by agglomeration techniques. See J. E. Browning, "Agglomeration",

Chemical Engineering, December 4, 1967, pages 147 and following, and "Perry's Chemical Engineer's Handbook", 4th Ed., McGraw-Hill, New York, 1963, pages 8 to 59 and following.

5

Example AEmulsifiable Concentrate

6,11-difluoro-3,3a,8,9-tetrahydro-N-[4-(trifluoromethoxy)phenyl]-2H-dibenzo[3,4:7,8]cyclooctan-

| | | |
|----|---------------------------------|-----|
| 10 | [1,2-c]pyrazole-2-carboxamide | 20% |
| | blend of oil soluble sulfonates | |
| | and polyoxyethylene ethers | 10% |
| | isophorone | 70% |

The ingredients are combined and stirred with gentle warming to speed solution. A fine screen filter is included in packaging operation to insure the absence of any extraneous undissolved material in the product.

15

Example B20 Wettable Powder

2-(1,9-difluoro-11,12-dihydrodibenzo[a,e]-cycloocten-5-(6H)-ylidene)-N-[4-(trifluoromethoxy)phenyl]hydrazine-2-carboxamide

| | | |
|----|----------------------------------|-----|
| | | 30% |
| | sodium alkylnaphthalenesulfonate | 2% |
| 25 | sodium ligninsulfonate | 2% |
| | synthetic amorphous silica | 3% |
| | kaolinite | 63% |

The active ingredient is mixed with the inert materials in a blender. After grinding in a hammer-mill, the material is re-blended and sifted through a 50 mesh screen.

30

145

Example CDust

- | | | |
|--|------------------------------|-----|
| | Wettable powder of Example B | 10% |
| | pyrophyllite (powder) | 90% |
- 5 The wettable powder and the pyrophyllite diluent are thoroughly blended and then packaged. The product is suitable for use as a dust.

Example D10 Granule

- 11-chloro-6-fluoro-3a,8-dihydro-N-[4-(tri-fluoromethoxy)phenyl]-3H-dibenzo[b,f]-pyrazolo[2,3-d][1,4]-oxazocine-2-carboxamide
- | | | |
|--|--|-----|
| | | 10% |
|--|--|-----|
- 15 attapulgite granules (low volatile matter, 0.71/0.30 mm; U.S.S. No. 25-50 sieves)
- | | | |
|--|--|-----|
| | | 90% |
|--|--|-----|

- The active ingredient is dissolved in a volatile solvent such as acetone and sprayed upon dedusted and
- 20 pre-warmed attapulgite granules in a double cone blender. The acetone is then driven off by heating. The granules are then allowed to cool and are packaged.

Example E25 Granule

- | | | |
|--|------------------------------|-----|
| | Wettable powder of Example B | 15% |
| | gypsum | 69% |
- 30 potassium sulfate
- | | | |
|--|--|-----|
| | | 16% |
|--|--|-----|
- The ingredients are blended in a rotating mixer and water sprayed on to accomplish granulation. When most of the material has reached the desired range of 0.1 to 0.42 mm (U.S.S. No. 18 to 40 sieves), the granules are

removed, dried, and screened. Oversize material is crushed to produce additional material in the desired range. These granules contain 4.5% active ingredient.

5

Example FSolution

2-fluoro-3,4,4a,9-tetrahydro-N-[4-(trifluoro-
methoxy)phenyl]-11-(trifluoromethyl)dibenzo-
[b,f]-pyridazino[2,3-d-][1,4]oxazocine-2-car-
boxamide 25%
N-methyl-pyrrolidone 75%

10

The ingredients are combined and stirred to produce a solution suitable for direct, low volume application.

15

Example GAqueous Suspension

6,11-difluoro-3,3a,8,9-tetrahydro-N-[4-(tri-
fluoro-methoxy)phenyl]-2H-dibenzo[3,4:7,8]-
cyclooctan-[1,2-c]pyrazole-2-carboxamide 40%
polyacrylic acid thickener 0.3%
dodecyclophenol polyethylene glycol
ether 0.5%
disodium phosphate 1.0%
monosodium phosphate 0.5%
polyvinyl alcohol 1.0%
water 56.7%

20

25

The ingredients are blended and ground together in a sand mill to produce particles substantially all under 5 microns in size.

30

Example HOil Suspension

- 2-(1,9-difluoro-11,12-dihydrodibenzo[a,e]-
cycloocten-5-(6H)-ylidene)-N-[4-(trifluoro-
5 methoxy)phenyl]hydrazine-2-carboxamide 35.0%
blend of polyalcohol carboxylic 6.0%
esters and oil soluble petroleum
sulfonates
xylene range solvent 59.0%
- 10 The ingredients are combined and ground together in
a sand mill to produce particles substantially all below
5 microns. The product can be used directly, extended
with oils, or emulsified in water.

15 Example IBait Granules

- 11-chloro-6-fluoro-3a,8-dihydro-N-[4-(tri-
fluoro-methoxy)phenyl]-3H-dibenzo[b,f]-
pyrazolo-[2,3-d][1,4]-oxazocine-
20 2-carboxamide 3.0%
blend of polyethoxylated nonyl- 9.0%
phenols and sodium dodecyl-
benzene sulfonates
ground up corn cobs 88.0%
- 25 The active ingredient and surfactant blend are
dissolved in a suitable solvent such as acetone and
sprayed onto the ground corn cobs. The granules are then
dried and packaged.
- 30 Compounds of Formula I can also be mixed with one or
more other insecticides, fungicides, nematocides,
bactericides, acaricides, or other biologically active
compounds to form a multi-component pesticide giving an
even broader spectrum of effective agricultural
protection. Examples of other agricultural protectants

with which compounds of this invention can be formulated are:

Insecticides:

- 5 3-hydroxy-N-methylcrotonamide (dimethylphosphate) ester
(monocrotophos)
methylcarbamic acid, ester with 2,3-dihydro-2,2-
dimethyl-7-benzofuranol (carbofuran)
O-[2,4,5-trichloro- α -(chloromethyl)benzyl]phosphoric
10 acid, O',O'-dimethyl ester (tetrachlorvinphos)
2-mercaptosuccinic acid, diethyl ester, S-ester with
thionophosphoric acid, dimethyl ester (malathion)
phosphorothioic acid, O,O-dimethyl, O-p-nitrophenyl
ester (methyl parathion)
15 methylcarbamic acid, ester with α -naphthol (carbaryl)
methyl O-(methylcarbamoyl)thiolacetohydroxamate
(methomyl)
N'-(4-chloro-p-tolyl)-N,N-dimethylformamidine
(chlordimeform)
20 O,O-diethyl-O-(2-isopropyl-4-methyl-6-pyrimidylphos-
phorothioate (diazinon)
octachlorocamphene (toxaphene)
O-ethyl-O-p-nitrophenyl phenylphosphonothioate (EPN)
(S)- α -cyano-m-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-
25 2,2-dimethylcyclopropanecarboxylate (deltamethrin)
Methyl-N',N'-dimethyl-N-[(methylcarbamoyl)oxy]-1-
thioox amimide (oxamyl)
cyano(3-phenoxyphenyl)-methyl-4-chloro-a-(1-methyl-
ethyl)benzeneacetate (fenvalerate)
30 (3-phenoxyphenyl)methyl (\pm)-cis,trans-3-(2,2-dichloro
ethenyl)-2,2-dimethylcyclopropanecarboxylate (perme-
thrin)
 α -cyano-3-phenoxybenzyl 3-(2,2-dichlorovinyl)-2,2-
dimethylcyclopropane carboxylate (cypermethrin)

- O-ethyl-S-(p-chlorophenyl)ethylphosphonodithioate
(profenofos)
phosphorothiolothionic acid,
O-ethyl-O-[4-(methylthio)-phenyl]-S-n-propyl ester
5 (sulprofos).

- Additional insecticides are listed hereafter by
their common names: triflumuron, diflubenzuron,
methoprene, buprofezin, thiodicarb, acephate, azinphos-
10 methyl, chlorpyrifos, dimethoate, fonophos, isofenphos,
methidathion, methamidphos, monocrotophos, phosmet,
phosphamidon, phosalone, pirimicarb, phorate, terbufos,
trichlorfon, methoxychlor, bifenthrin, biphenate,
cyfluthrin, fenpropathrin, fluvalinate, flucythrinate,
15 tralomethrin, metaldehyde and rotenone.

Fungicides:

- methyl 2-benzimidazolecarbamate (carbendazim)
tetramethylthiuram disulfide (thiuram)
20 n-dodecylguanidine acetate (dodine)
manganese ethylenebisdithiocarbamate (maneb)
1,4-dichloro-2,5-dimethoxybenzene (chloroneb)
methyl 1-(butylcarbamoyl)-2-benzimidazolecarbamate
(benomyl)
25 1-[2-(2,4-dichlorophenyl)-4-propyl-1,3-dioxolan-
2-ylmethyl]-1H-1,2,4-triazole (propiconazole)
2-cyano-N-ethylcarbamoyl-2-methoxyiminoacetamide
(cymoxanil)
1-(4-chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazol-
30 1-yl)-2-butanone (triadimefon)
N-(trichloromethylthio)tetrahydrophthalimide (captan)
N-(trichloromethylthio)phthalimide (folpet)
1-[[[bis(4-fluorophenyl)][methyl]silyl]methyl]-1H-
1,2,4-triazole.

Nematocides:

- S-methyl 1-(dimethylcarbamoyl)-N-(methylcarbamoyloxy)-
thioformimidate
- 5 S-methyl 1-carbamoyl-N-(methylcarbamoyloxy)-
thioformimidate
- N-isopropylphosphoramidic acid O-ethyl O'-[4-(methyl-
thio)-m-tolyl]diester (fenamiphos)

10 Bactericides:

tribasic copper sulfate
streptomycin sulfate

Acaricides:

- 15 senecioic acid, ester with 2-sec-butyl-4,6-dinitro-
phenol (binapacryl)
- 6-methyl-1,3-cithiolo[4,5-B]quinoxalin-2-one
(oxythioquinox)
- ethyl 4,4'-dichlorobenzilate (chlorobenzilate)
- 20 1,1-bis(p-chlorophenyl)-2,2,2-trichloroethanol
(dicofol)
- bis(pentachloro-2,4-cyclopentadien-1-yl) (dienochlor)
- tricyclohexyltin hydroxide (cyhexatin)
- trans-5-(4-chlorophenyl)-N-cyclohexyl-4-methyl-2-oxo-
- 25 thiazolidine-3-carboxamide (hexythiazox)
- amitraz
- propargite
- fenbutatin-oxide

30 Biological

Bacillus thuringiensis
Avermectin B.

Utility

The compounds of this invention exhibit activity against a wide spectrum of foliar and soil inhabiting arthropods which are pests of growing and stored

5 agronomic crops, forestry, greenhouse crops, ornamentals, nursery crops, stored food and fiber products, livestock, household, and public and animal health. Those skilled in the art will recognize that not all compounds are equally effective against all pests but the compounds of

10 this invention display activity against economically important agronomic, forestry, greenhouse, ornamental food and fiber product, stored product, domestic structure, and nursery pests, such as:

15 larvae of the order Lepidoptera including fall and beet armyworm and other Spodoptera spp., tobacco budworm, corn earworm and other Heliothis spp., European corn borer, navel orangeworm, stalk/stem borers and other

20 pyralids, cabbage and soybean loopers and other loopers, codling moth, grape berry moth and other tortricids, black cutworm, spotted cutworm, other cutworms and other noctuids, diamondback moth, green cloverworm, velvetbean

25 caterpillar, green cloverworm, pink bollworm, gypsy moth, and spruce budworm;

foliar feeding larvae and adults of the order Coleoptera including Colorado potato beetle,

30 Mexican bean beetle, flea beetle, Japanese beetles, and other leaf beetles, boll weevil, rice water weevil, granary weevil, rice weevil and other weevil pests, and soil inhabiting insects such as Western corn rootworm and other

Diabrotica spp., Japanese beetle, European chafer and other coleopteran grubs, and wireworms;

5 adults and larvae of the orders Hemiptera and Homoptera including tarnished plant bug and other plant bugs (miridae), aster leafhopper and other leafhoppers (cicadellidae), rice planthopper, brown planthopper, and other
10 planthoppers (fulgoroidea), psyllids, whiteflies (aleurodidae), aphids (aphidae), scales (coccidae and diaspididae), lace bugs (tingidae), stink bugs (pentatomidae), cinch bugs and other seed bugs (lygaeidae), cicadas
15 (cicadidae), spittlebugs (cercopids), squash bugs (coreidae), red bugs and cotton stainers (pyrrhocoridae);

20 adults and larvae of the order acari (mites) including European red mite, two spotted spider mite, rust mites, McDaniel mite, and foliar feeding mites;

25 adults and immatures of the order Orthoptera including grasshoppers;

adults and immatures of the order Diptera including leafminers, midges, fruit flies (tephritidae), and soil maggots;

30 adults and immatures of the order Thysanoptera including onion thrips and other foliar feeding thrips.

The compounds are also active against economically important livestock, household, public and animal health pests such as:

- 5 insect pests of the order Hymenoptera including
carpenter ants, bees, hornets, and wasps;
- 10 insect pests of the order Diptera including
house flies, stable flies, face flies, horn
flies, blow flies, and other muscoid fly pests,
horse flies, deer flies and other Brachycera,
mosquitoes, black flies, biting midges, sand
flies, sciarids, and other Nematocera;
- 15 insect pests of the order Orthoptera including
cockroaches and crickets;
- 20 insect pests of the order Isoptera including the
Eastern subterranean termite and other termites;
insect pests of the order Mallophaga and
Anoplura including the head louse, body louse,
chicken head louse and other sucking and chewing
parasitic lice that attack man and animals;
- 25 insect pests of the order Siphonoptera including
the cat flea, dog flea and other fleas.

The specific species for which control is
exemplified are: fall armyworm, Spodoptera frugiperda;
30 tobacco budworm, Heliothis virescens; southern corn
rootworm, Diabrotica undecimpunctata. The pest control
protection afforded by the compounds of the present
invention is not limited, however, to these species. The

compounds of this invention may also be utilized as rodenticides.

Application

5 Arthropod pests are controlled and protection of
agronomic crops, animal and human health is achieved by
applying one or more of the Formula I compounds, in an
effective amount, to the environment of the pests
including the agronomic and/or nonagronomic locus of
10 infestation, to the area to be protected, or directly on
the pests to be controlled. Because of the diversity of
habitat and behavior of these arthropod pest species,
many different methods of application are employed. A
preferred method of application is by spraying with
15 equipment that distributes the compound in the
environment of the pests, on the foliage, animal, person,
or premise, in the soil or animal, to the plant part that
is infested or needs to be protected. Alternatively,
granular formulations of these toxicant compounds can be
20 applied to or incorporated into the soil. Other methods
of application can also be employed including direct and
residual sprays, aerial sprays, baits, eartags, boluses,
foggers, aerosols, and many others. The compounds can be
incorporated into baits that are consumed by the
25 arthropods or in devices such as traps and the like which
entice them to ingest or otherwise contact the compounds.

The compounds of this invention can be applied in
their pure state, but most often application will be of a
formulation comprising one or more compounds with
30 suitable carriers, diluents, and surfactants and possibly
in combination with a food depending on the contemplated
end use. A preferred method of application involves
spraying a water dispersion or refined oil solution of
the compounds. Combinations with spray oils, spray oil

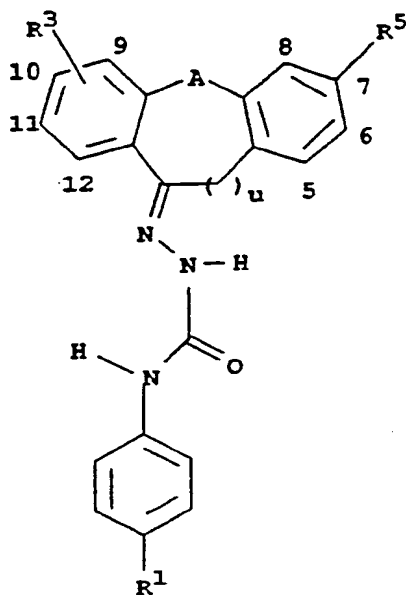
concentrations, and synergists such as piperonyl butoxide often enhance the efficacy of the compounds of Formula I.

5 The rate of application required for effective control will depend on such factors as the species of arthropod to be controlled, the pest's life cycle, life stage, its size, location, time of year, host crop or animal, feeding behavior, mating behavior, ambient moisture, temperature, etc. In general, application rates of 0.01 to 2 kg of active ingredient per hectare
10 are sufficient to provide large-scale effective control of pests in agronomic ecosystems under normal circumstances, but as little as 0.001 kg/hectare or as much as 8 kg/hectare may be required. For nonagronomic applications, effective use rates will range from about
15 1.0 to 50 mg/square meter but as little as about 0.1 mg/square meter or as much as 150 mg/square meter may be required.

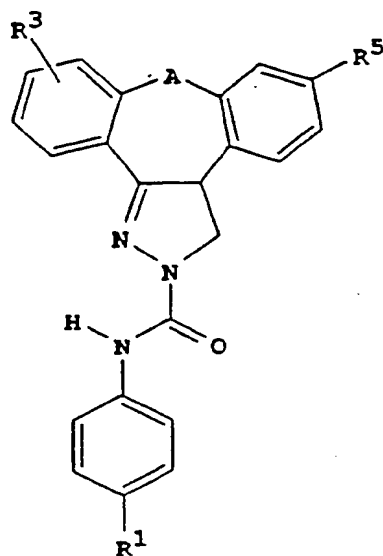
The following tests demonstrate the control efficacy
20 of a compound of Formula I on specific pests; see Index Table A for compound description.

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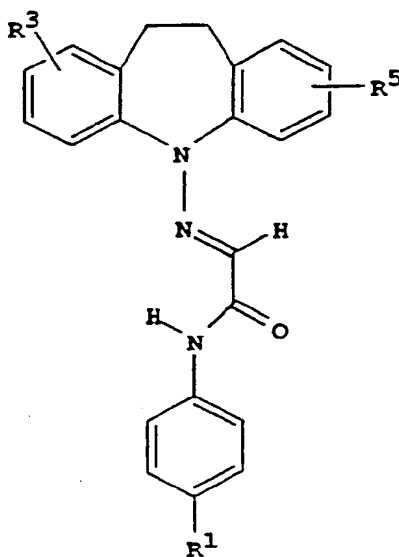
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INDEX TABLE A

| <u>COMPOUND</u> | <u>R¹</u> | <u>R³</u> | <u>R⁵</u> | <u>A</u> | <u>n</u> | <u>Melting Point (°C)</u> |
|-----------------|----------------------|----------------------|----------------------|---------------------------------|----------|---------------------------|
| 1 | OCF ₃ | H | H | CH ₂ CH ₂ | 0 | 172-173 |
| 2 | OCF ₃ | 9-F | H | OCH ₂ | 0 | 188-191 |
| 3 | CF ₃ | 9-F | H | OCH ₂ | 0 | 165-170 |

INDEX TABLE B

| <u>COMPOUND</u> | R^1 | R^3 | R^5 | A | <u>Melting Point ($^{\circ}\text{C}$)</u> |
|-----------------|---------------|-------|-------|--------------------------|--|
| 4 | CF_3 | H | H | CH_2CH_2 | 245-250 |
| 5 | Br | H | H | CH_2CH_2 | 188-191 |

INDEX TABLE C

| <u>COMPOUND</u> | R^1 | R^3 | R^5 | <u>Melting Point ($^{\circ}C$)</u> |
|-----------------|--------|-------|-------|---|
| 6 | CF_3 | H | H | 80-85 |

Insecticide Test ProtocolsCompound Application

The experimental compound is formulated in a 75:25 acetone:water solution, unless otherwise indicated. The compound is initially tested at 1000 ppm. The formulated compound is applied with a single, flat fan 8001E nozzle positioned 7.5 inches (19 cm) above the test units which are situated on a conveyor belt. Spray pressure is maintained at 30 psi (207 kPa), and the conveyor speed is adjusted so that 6 ml of test solution is sprayed per 0.1 square meter of conveyor at a rate of 0.5 pounds (0.2 kg) of active ingredient per acre (0.55 kg/ha). Three untreated (blanks) and three solvent-treated test units are run for each insect species tested.

15

EXAMPLE JFall Armyworm (FAW) Spodoptera frugiperda

Acute Toxicity: Two lima bean leaf discs, each with a surface area of 8.1 cm² were sprayed top side up along with 7-12 3rd instar, unstarved fall armyworm larvae. The treated lima bean leaves were placed top side up in a 15 mm x 100 mm petri dish that had been lined with filter paper moistened with 1.5 ml of water. After the leaf discs had dried, 5 sprayed larvae were introduced into the petri dish. Larval mortality was assessed at 48 hours post-treatment. The following compounds exhibited mortality of 80% or greater: 1, 2 and 3.

20

25

EXAMPLE K

Tobacco Budworm (TBW)

Heliothis virescens (helicoverpa)

Five 3rd instar larvae were placed in an 8 oz (230 ml) cup containing artificial diet and sprayed with the test solution. Larval mortality was assessed at 48 hours post-treatment. The following compounds exhibited mortality of 80% or greater: 1, 2 and 3.

30

EXAMPLE L

Southern Corn Rootworm (SCRW)

Diabrotica undecimpunctata howardi

5 An 8 oz (230 ml) dish containing a germinated corn
kernel was sprayed with the test solution. After the
spray had dried, five unsprayed, 3rd instar corn rootworm
larvae were placed in the dish along with a moistened
cotton wick. Larval mortality was assessed at 48 hours
post-treatment. The following compounds exhibited
10 mortality of 80% or greater: 1, 2 and 3.

EXAMPLE M

Boll Weevil (BW)

Anthonomus grandis grandis

15 Five adult boll weevils were placed into a 9 ounce
(260 mL) cup containing artificial diet and sprayed.
Mortality readings were taken 48 hours after treatment.
The following compounds exhibited mortality of 80% or
greater: 2 and 3.

20

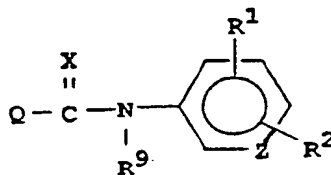
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CLAIMS

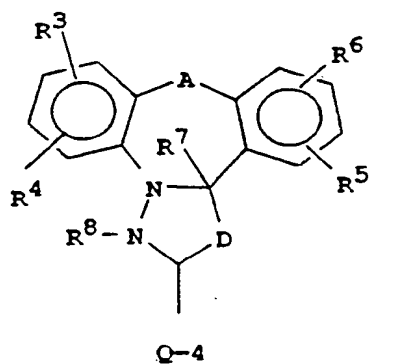
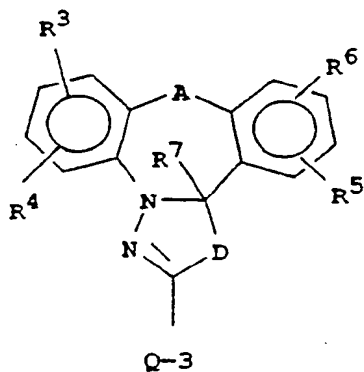
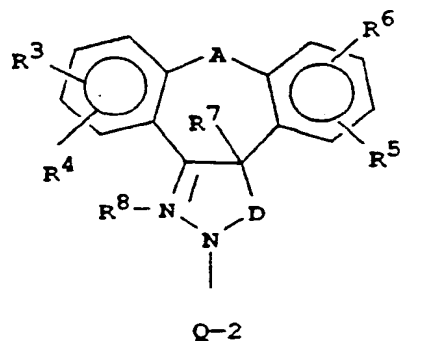
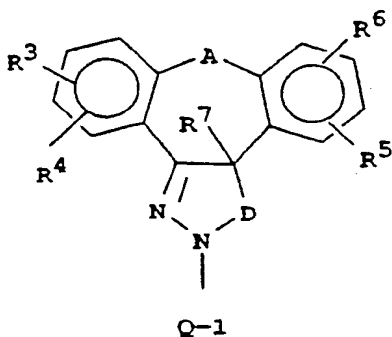
What is claimed is:

- 5 1. A compound of the formula

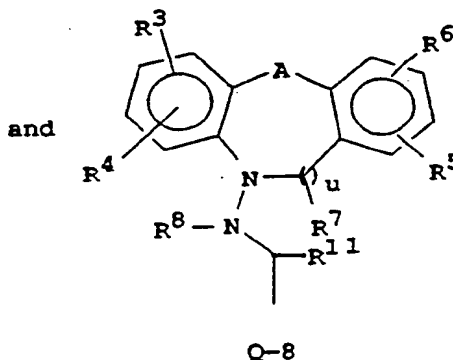
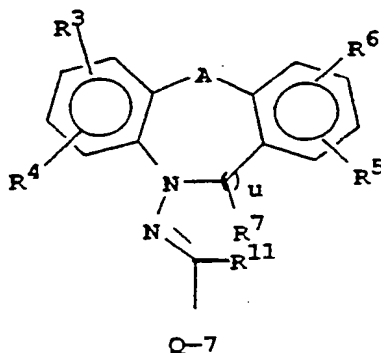
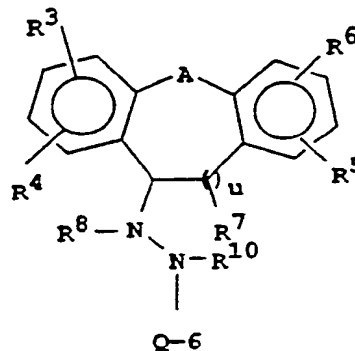
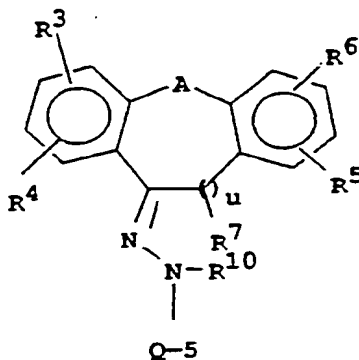


wherein:

- 10 Q is selected from the group



15



- 5 A is selected from the group CH_2 , CH_2CH_2 , O, $\text{S}(\text{O})_p$, NR^{18} and $-\text{CH}_2(\text{G})-$; provided that i) when A is $-\text{CH}_2(\text{G})-$ either phenyl moiety can be attached to the CH_2 moiety and when A is CH_2 or $-\text{CH}_2(\text{G})-$, said CH_2 can be optionally substituted with 1 or
- 10 2 substituents independently selected from C_1 - C_2 alkyl, and ii) when u is 0, A is CH_2CH_2 or $-\text{CH}_2(\text{G})-$;
- D is selected from C_1 - C_2 alkylene optionally substituted with 1 to 2 substituents
- 15 independently selected from C_1 - C_2 alkyl;
- G is selected from the group O, $\text{S}(\text{O})_p$ and NR^{18} ;
- X is selected from the group O and S;
- Z is selected from the group N and CH;
- R^1 , R^2 , R^3 , R^4 , R^5 and R^6 are independently selected
- 20 from the group H, halogen, CN, SCN, NO_2 , R^{12} ,

- OR¹², S(O)_qR¹², OSO₂R¹², C(O)R¹², CO₂R¹²,
C(O)N(R¹²)R¹³, SO₂N(R¹²)R¹³ and N(R¹²)R¹³; or
R¹ and R² or R³ and R⁴ or R⁵ and R⁶ when attached to
adjacent atoms can be taken together as OCH₂O,
5 OCF₂O, OCH₂CH₂O, OCH₂C(CH₃)₂O or OCF₂CF₂O to
form a cyclic bridge;
R⁷ is selected from the group H, CN, C₁-C₆ alkyl and
CO₂R¹²;
R⁸ is selected from the group H, C₁-C₆ alkyl, C₂-C₆
10 alkylcarbonyl, CO₂R¹² and C(O)N(R¹²)R¹³;
R⁹ and R¹⁰ are independently selected from the group
H, C₁-C₆ alkyl, C₂-C₆ alkoxyalkyl, CHO, C₂-C₆
alkylcarbonyl, C₂-C₆ alkoxycarbonyl,
C₂-C₆ haloalkylcarbonyl, C₁-C₆ haloalkylthio,
15 R¹⁴OC(O)N(R¹⁵)S-, R¹⁷(R¹⁶)NS- and benzyl
optionally substituted with W;
R¹¹ is selected from the group H, C₁-C₆ alkyl,
C₁-C₆ haloalkyl and phenyl optionally substituted
with W;
20 R¹² is selected from the group C₁-C₄ alkyl, C₁-C₄
haloalkyl, C₂-C₄ alkenyl, C₂-C₄ haloalkenyl, C₃-
C₄ alkynyl, C₃-C₄ haloalkynyl, C₂-C₆
alkoxyalkyl, C₂-C₆ alkylthioalkyl, C₂-C₆
cyanoalkyl, C₃-C₆ alkoxycarbonylalkyl, C₃-C₆
25 cycloalkyl, C₃-C₆ halocycloalkyl, C₄-C₇
alkylcycloalkyl, C₄-C₇ haloalkylcycloalkyl,
optionally substituted phenyl and optionally
substituted benzyl wherein the phenyl and benzyl
substituent(s) are 1 to 3 substituents
30 independently selected from W;
R¹³ is selected from the group H and C₁-C₄ alkyl;
R¹⁴ and R¹⁵ are independently selected from C₁-C₆
alkyl;

R¹⁶ and R¹⁷ are independently selected from C₁-C₄ alkyl; or
R¹⁶ and R¹⁷ when attached to the same atom can be taken together as (CH₂)₅ or CH₂CH₂OCH₂CH₂;
5 R¹⁸ is selected from the group H, C₁-C₃ alkyl, CO₂R¹⁹ and SO₂R¹⁹;
R¹⁹ is selected from C₁-C₃ alkyl;
W is selected from the group halogen, CN, NO₂,
C₁-C₂ alkyl, C₁-C₂ haloalkoxy, C₁-C₂ alkoxy, C₁-
10 C₂ haloalkoxy, C₁-C₃ alkylthio, C₁-C₂ haloalkylthio, C₁-C₂ alkylsulfonyl and C₁-C₂ haloalkylsulfonyl;
p is 0, 1 or 2;
q is 0, 1 or 2; and
15 u is 0 or 1.

2. A compound according to Claim 1 wherein:
A is selected from the group S, CH₂CH₂ and -CH₂(G)-;
20 D is C₁-C₂ alkylene;
R¹, R², R³, R⁴, R⁵ and R⁶ are independently selected from the group H, halogen, CN, R¹², S(O)_qR¹² and OSO₂R¹²;
R⁷ is CH₃;
25 R⁸ is H;
R⁹ and R¹⁰ are independently selected from the group H, C₁-C₂ alkyl, C₂-C₃ alkylcarbonyl and C₂-C₃ alkoxy carbonyl;
R¹¹ is selected from the group H and CH₃;

R¹² is selected from the group C₁-C₃ alkyl and
C₁-C₃ haloalkyl;
R¹³ is C₁-C₂ alkyl;
R¹⁸ is H or CH₃;
p is 0; and
q is 0 or 2.

5

3. A compound according to Claim 2 wherein Q is
Q-1.

10

4. A compound according to Claim 2 wherein Q is
Q-2.

15

5. A compound according to Claim 2 wherein Q is
Q-3.

6. A compound according to Claim 2 wherein Q is
Q-4.

20

7. A compound according to Claim 2 wherein Q is
Q-5.

25

8. A compound according to Claim 2 wherein Q is
selected from Q-6, Q-7 and Q-8.

9. An arthropodicidal composition comprising a
compound according to any one of Claims 1 to 8 and a
carrier therefor.

30

10. A method for controlling arthropods comprising
contacting them or their environment with an
arthropodically effective amount of a compound
according to any one of Claims 1 to 8.



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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|---|-----------------------------|---|---------------------------|----|---------|-----------------------------|----|----|---------------|----------|---------------------------|----|---------------|----------|-----------------------------|---|
| <p>(51) International Patent Classification ⁵ :</p> <p>C07D 231/54, 223/20, 237/26 C07D 267/22, 313/12, 313/20 C07D 337/14</p> | <p>A3</p> | <p>(11) International Publication Number: WO 92/12133</p> <p>(43) International Publication Date: 23 July 1992 (23.07.92)</p> | | | | | | | | | | | | | | |
| <p>(21) International Application Number: PCT/US91/09172</p> <p>(22) International Filing Date: 17 December 1991 (17.12.91)</p> <p>(30) Priority data:</p> <table border="0"> <tr> <td>638,720</td> <td>8 January 1991 (08.01.91)</td> <td>US</td> </tr> <tr> <td>657,702</td> <td>19 February 1991 (19.02.91)</td> <td>US</td> </tr> </table> <p>(60) Parent Applications or Grants</p> <p>(63) Related by Continuation</p> <table border="0"> <tr> <td>US</td> <td>638,720 (CIP)</td> </tr> <tr> <td>Filed on</td> <td>8 January 1991 (08.01.91)</td> </tr> <tr> <td>US</td> <td>657,702 (CIP)</td> </tr> <tr> <td>Filed on</td> <td>19 February 1991 (19.02.91)</td> </tr> </table> <p>(71) Applicant (for all designated States except US): E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).</p> | | 638,720 | 8 January 1991 (08.01.91) | US | 657,702 | 19 February 1991 (19.02.91) | US | US | 638,720 (CIP) | Filed on | 8 January 1991 (08.01.91) | US | 657,702 (CIP) | Filed on | 19 February 1991 (19.02.91) | <p>(72) Inventors; and</p> <p>(75) Inventors/Applicants (for US only): HARRISON, Charles, Richard [US/US]; 137 Aspen Drive, Newark, DE 19702 (US). KRANIS, Kevin, Thomas [US/US]; 1413 North 12th Street, Reading, PA 19604 (US). STEVENSON, Thomas, Martin [US/US]; 103 Iroquois Court, Newark, DE 19702 (US).</p> <p>(74) Agents: COSTELLO, James, A. et al.; E.I. du Pont de Nemours and Company, Legal/Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).</p> <p>(81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), MC (European patent), NL (European patent), SE (European patent), US.</p> <p>Published</p> <p><i>With international search report.</i></p> <p><i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p> <p>(88) Date of publication of the international search report: 15 October 1992 (15.10.92)</p> |
| 638,720 | 8 January 1991 (08.01.91) | US | | | | | | | | | | | | | | |
| 657,702 | 19 February 1991 (19.02.91) | US | | | | | | | | | | | | | | |
| US | 638,720 (CIP) | | | | | | | | | | | | | | | |
| Filed on | 8 January 1991 (08.01.91) | | | | | | | | | | | | | | | |
| US | 657,702 (CIP) | | | | | | | | | | | | | | | |
| Filed on | 19 February 1991 (19.02.91) | | | | | | | | | | | | | | | |
| <p>(54) Title: ARTHROPODICAL CARBOXANILIDES</p> <div style="text-align: center; margin: 20px 0;"> <p style="margin-top: 10px;">(I)</p> </div> <p>(57) Abstract</p> <p>Compounds of formula (I), wherein Q, X, R¹, R², R⁹ and Z are as defined in the text, including compositions containing said compounds and a method for using them to control arthropods.</p> | | | | | | | | | | | | | | | | |

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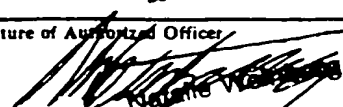
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INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 91/09172

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|--|--|-------------------------------------|
| I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶ | | |
| According to International Patent Classification (IPC) or to both National Classification and IPC | | |
| Int.Cl. 5 | C 07 D 231/54 | C 07 D 223/20 |
| C 07 D 267/22 | C 07 D 313/12 | C 07 D 313/20 |
| | | C 07 D 237/26 |
| | | C 07 D 337/14 |
| II. FIELDS SEARCHED | | |
| Minimum Documentation Searched ⁷ | | |
| Classification System | Classification Symbols | |
| Int.Cl. 5 | C 07 D | A 01 N |
| | | C 07 C |
| Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸ | | |
| III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹ | | |
| Category ¹⁰ | Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹² | Relevant to Claim No. ¹³ |
| X | EP,A,0312051 (KYOWA HAKKO KOGYO CO., LTD) 19 April 1989, see page 4, line 47 - page 5, line 26 --- | 1,2,7,8 |
| A | US,A,4198421 (E.I. DU PONT DE NEMOURS AND COMPANY) 15 April 1980 --- | |
| A | Chemical Abstracts, volume 86, no. 5, 31 January 1977 (Columbus, Ohio, US) see page 355, abstract 29670e & JP, A, 7665762 (JAPAN CHEMIPHA CO., LTD) 7 June 1976 --- | |
| A | Chemical Abstracts, volume 86, no. 25, 20 June 1977, (Columbus, Ohio, US) see page 593, abstract 189744k, & JP, A, 76108082 (JAPAN CHEMIPHA CO., LTD) 25 September 1976 --- | |
| | -/- | |
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| IV. CERTIFICATION | | |
| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report | |
| 14-04-1992 | 18.09.92 | |
| International Searching Authority | Signature of Authorized Officer | |
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III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

| Category * | Citation of Document, with indication, where appropriate, of the relevant passages | Relevant to Claim No. |
|------------|---|-----------------------|
| A | Chemical Abstracts, volume 86, no. 25, 20 June 1977 (Columbus, Ohio, US) see page 593, abstract 189745m, & JP, A, 76108081 (JAPAN CHEMIPHA CO., LTD) 25 September 1976 --- | |
| A | EP,A,0189310 (SPOFA) 30 July 1986 ----- | |

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ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.

US 9109172

SA 55972

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| | | US-A- 5010104 | 23-04-91 |
| | | US-A- 5010087 | 23-04-91 |
| US-A- 4198421 | 15-04-80 | None | |
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82